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Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity

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CANADIAN DEVELOPMENTS IN TESTING CONCRETE AGGREGATES FOR ALKALI-AGGREGATE REACTIVITY

Editor:	C.A. ROGERS
Abstract:	This document contains papers presented at a conference held in Toronto on March 19, 1990. Alkali-aggregate reactions occur in Portland cement concrete between the cement alkalies, sodium and potassium hydroxide, and the aggregates. These reactions can lead to expansion and cracking of concrete which reduces strength and allows increased amounts of water and de-icing chemicals to enter the concrete. These reactions eventually result in increased repair costs of damaged structures and pavements and ultimately reduce their life. There is no satisfactory cure for these reactions, thus it is necessary to avoid reactive aggregates by laboratory testing prior to construction. The laboratory testing is difficult and time consuming. Over the past few years, Canadian researchers have been experimenting with new, rapid test methods. The purpose of the meeting in Toronto was to provide a forum for presentation of research findings and to bring this problem to the attention of Canadian users and makers of concrete and concrete products. It is hoped to introduce some of the results of this research in future revisions of Canadian Standards Association and provincial specifications for concrete aggregates.
Résumé:	Le présent document contient divers articles présentés lors d'une conférence ayant eu lieu à Toronto le 19 mars 1990. Des réactions alcalis-agrégats surviennent dans le ciment Portland entre les alcalis du ciment, le sodium et l'hydroxyde de potassium d'une part et les agrégats, d'autre part. Ces réactions peuvent entraîner la dilatation, le craquement et l'affaiblissement du béton et permettre à une plus grande quantité d'eau et de produits chimiques de dégivrage de pénétrer dans le béton. Elles peuvent également se traduire par une hausse des coûts de réparation des structures et des chaussées et, éventuellment, par une réduction de la durée de vie de ces dernières. On ne peut empêcher ces réactions; il est donc nécessaire d'éviter l'utilisation d'agrégats réactifs en analysant les matériaux en laboratoire avant le début des travaux de construction. Ces essais en laboratoire sont difficiles et prennent beaucoup de temps. Au cours des dernières années, les chercheurs canadiens ont expérimenté de nouvelles méthodes d'essai rapides. La conférence de Toronto visait à présenter le fruits de ces travaux de recherche et à porter ce problème à l'attention des utilisateurs et des fabricants canadiens de béton et de produits de béton. On espère que certains des ces résultats seront intégrés aux modifications futures apportées aux spécifications provinciales et à celles de l'Association canadienne de normalisation relatives aux agrégats de béton.
Keywords:	aggregates, performance, petrographic examination, concrete, alkalies, Portland cement, testing, alkali-aggregate reaction, expansion, cracking.
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REPORT EM-92

CANADIAN DEVELOPMENTS IN TESTING CONCRETE AGGREGATES FOR ALKALI-AGGREGATE REACTIVITY

edited by: C. A. Rogers

Papers presented at a conference on "Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity", Toronto, March 1990. Sponsored by the Canadian Standards Association sub-committee on cement-aggregate reactivity (CSA-A5); American Concrete Institute, Ontario Chapter; and Ministry of Transportation, Ontario

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ALKALI-AGGREGATE REACTIVITY IN CANADA

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ABSTRACT

In Canada, three types of alkali-aggregate reaction are recognized. Each type is evaluated using different tests. Corrective measures such as the use of low alkali cement, lower cement contents, or pozzolans are seldom used with reactive aggregates. Beneficiation or selective extraction is used with some reactive aggregates. Work is being conducted on multilaboratory study of existing tests and new, rapid tests.

INTRODUCTION

The oldest Canadian concrete structure known to have been affected by alkali-aggregate reactivity (A.A.R.) was the Hurdman Bridge built in Ottawa in 1906 and demolished in 1987. It was not until the 1950's, however, that A.A.R. was first documented in Canada (Swenson, 1957). Over the past thirty years, cases of A.A.R. have been found in many parts of Canada (Figures 1 and 2) with a variety of different rock types (Table 1). There are now over 160 reports and papers documenting Canadian work on A.A.R. (Rogers and Worton, 1988).

The wide range in rock types capable of causing damage due to A.A.R. gives some idea of the difficulty of selecting appropriate testing procedures and corrective measures. For instance, the mortar bar expansion test used for alkali-silica reactive rocks only gives small expansions with expansive dolomitic limestone susceptible to the alkali-carbonate reaction (A.C.R.) or greywackes and argillites susceptible to the alkali-silicate reaction. For convenience, alkali-aggregate reactions in Canada have been grouped into three types, each having different tests or criteria used in evaluation:

1. Alkali-carbonate reaction with dolomitic limestones and calcitic dolostones of Ordovician age. Chemical analysis for CaO:MgO and Al_2O_3, rock cylinder expansion test (ASTM C 586), and concrete prism expansion test (CAN3 A23.2-14A, 1986) are useful for identifying these rocks.

TABLE 1: Types of Alkali-Reactive Rock Found in Canada

Phanerozoic
Andesites and associated volcanic rocks in British Columbia
Quartzite in Rocky Mountains of British Columbia and Alberta
Opaline shale/siliceous shale in Saskatchewan and Manitoba
Upper Silurian subgreywacke on Ellesmere Island
Devonian and Silurian chalcedony-bearing chert in glacial drift in northern Ontario and in quarries and glacial drift in southern Ontario
Middle Ordovician siliceous and slightly siliceous limestone in southern and eastern Ontario and the St. Lawrence Lowlands of Quebec
Middle Ordovician dolomitic limestone and calcitic dolostone in Hudson Bay Lowlands and southern Ontario
Cambrian sandstones and orthoquartzites in eastern Ontario and southwestern Quebec
Rhyolitic tuff, southeast of Quebec City
Phyllite in southeastern Quebec
Phyllites, greywackes, siltstones, argillites, schists, silicified tuff, devitrified glass, and quartzites of New Brunswick, Nova Scotia, and Newfoundland
Rhyolite in Nova Scotia
Precambrian
Granite and granite gneiss with strained quartz
Sandstone, quartz-wacke, arkose, greywacke, and argillite of the Huronian Supergroup in central Ontario and western Quebec
Chert in banded iron formations (taconite)

2. Alkali-silica reaction with silica (chalcedony, opal, cristobalite, etc.) is usually evaluated in the mortar bar expansion test (ASTM C 227); concrete prism and rock cylinder expansion tests are also used. The quick chemical test (ASTM C 289) is also used, but gives misleading results when carbonates are present.

3. Late/slow alkali-silica/silicate reaction with sandstones and granites containing strained quartz and metamorphosed sediments such as phyllite, argillite, and greywacke, where exfoliation of phyllosilicates causes swelling of aggregate particles. Rocks susceptible to this type of reaction usually perform well in conventional alkali-aggregate tests. A concrete prism expansion test at 38°C and high alkali contents is the most useful procedure. Rock cylinder or miniature rock prism expansion tests are also useful.

The procedures necessary to determine the type or types of reaction to which aggregate may be susceptible are not simple. Flow charts used for determining the type of reaction and the necessary tests were described in a paper at the 1986 conference (Rogers, 1987).

CANADIAN CEMENTS

Figures 1 and 2 show that the majority of areas of A.A.R. are in central and eastern Canada where low alkali cements are seldom used. West of Ontario, most of the normal Portland cements (CSA Type 10) have alkali levels of less than 0.8% (Na_2O equivalent) and, as a result, there are very few documented cases of A.A.R.

In central and eastern Canada, the majority of normal cements have alkali levels of more than 0.9%. For the nineteen normal cements made in Canada, the Na_2O/K_2O ratio ranges from 1.09 to 0.07 with a mean of 0.38 (median 0.31). In a 1977 survey of the alkali contents of Canadian Type 10 cements, six of twenty-three sources had alkali contents of more than 1.0% (Grattan-Bellew et al, 1978). In 1988, only two of nineteen producers had mean alkalies of more than 1.0%. This reduction in alkali contents can generally be attributed to the closing of obsolete plants that used high alkali raw materials.

At present, two normal Portland cements are made in Canada which contain a pozzolanic mineral admixture (Type 10P with 6 and 8% silica fume); the remainder are Portland cements with usually up to 5% limestone interground with the clinker.

Six plants (three in the east and three in western Canada) which normally make high alkali cements also make a lower alkali cement, usually without 5% limestone addition, to meet American requirements. Many state highway authorities adjacent to the Canadian border specify either a low alkali cement (<0.6%) or a reduced alkali cement (<0.7%). These lower alkali cements are usually reserved for the export market to the U.S., and not normally sold in Canada. In 1989, the Ministry of Transportation called for the use of a lower alkali cement (<0.7%) for concrete paving with a slightly reactive aggregate near Peterborough, Ontario. The paving subcontractor was unable to secure supplies of such cement, and proposed the use of non-reactive aggregates transported about 150 km.

The specification of low alkali cement is unusual in eastern Canada. In northern Ontario, the Ministry of Transportation used low alkali cement on four bridges in 1970, but has seldom specified it since. Recently, however, Canadian National Railways has started to specify low alkali cement for all concrete, irrespective of the nature of the aggregate. The reason for this was the costly and tedious deterioration of prestressed concrete railroad ties due to A.A.R. The cement companies regard the universal adoption of such a solution to A.A.R. with concern. The older, wet process plants can often make a low or reduced alkali cement by not recycling alkali-rich dust from the precipitators. The newer, dry process plants can only achieve a low alkali cement by selecting low alkali raw material sources. In either case, the cost of production is usually increased. There is a natural apprehension that other authorities or purchasers will begin to specify the universal use of low alkali cement to reduce the likelihood of A.A.R.

Figure 1: Location and Alkali Contents of Type 10 Cements in Eastern Canada

Figure 2: Location and Alkali Contents of Type 10 Cements in Western Canada

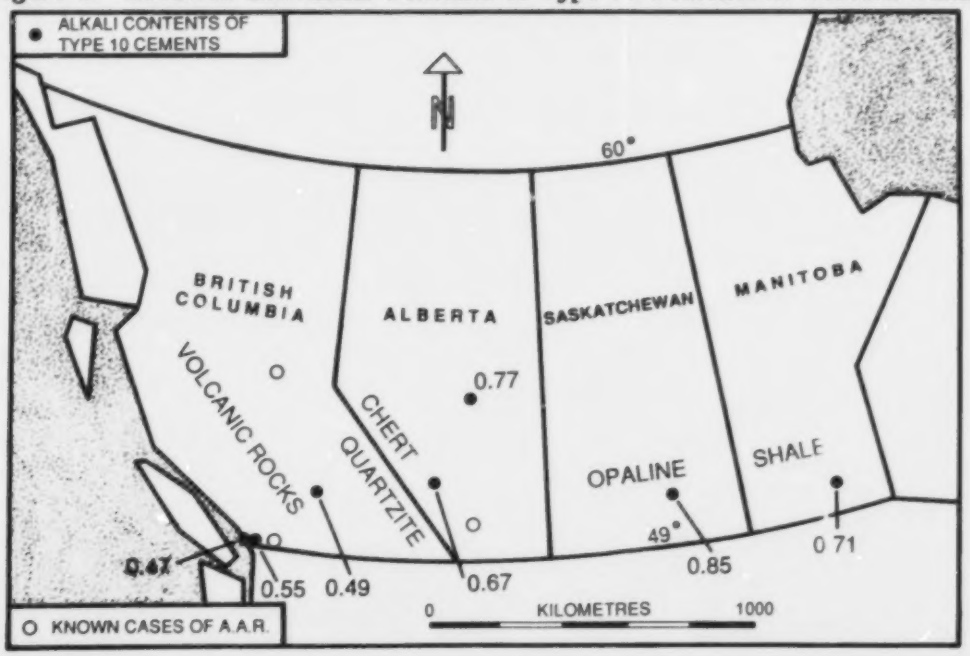

TABLE 2 - Alkali contents of Canadian Portland cements (latest monthy or yearly average, jan. 1989)

Cement Co.,	Location	Capacity Tonne x 10^6	Cement Type	Alkalies, Percent Na_2O	K_2O	Eq. Na_2O
TILBURY CEMENT	Delta, B.C.	1.25	10	0.28	0.29	0.47
LAFARGE CANADA	Richmond, B.C.	0.33	10	0.35	0.32	0.55
			3	0.33	0.20	0.49
			50	0.30	0.32	0.50
LAFARGE CANADA	Kamloops, B.C.	0.19	10	0.20	0.44	0.49
			50	0.20	0.50	0.53
LAFARGE CANADA	Exshaw, Alberta	1.10	10	0.15	0.79	0.67
			3	0.15	0.80	0.68
			50	0.15	0.68	0.60
INLAND CEMENT	Edmonton, Alberta	0.73	10	0.22	0.84	0.77
			50	0.24	0.76	0.74
	Low alkali to special order			0.20	0.47	0.51
INLAND CEMENT	Regina, Saskatchewan	0.20	10	0.32	0.80	0.85
			50	0.35	0.63	0.76
	Low alkali to special order			No Data.		
INLAND CEMENT	Winnipeg, Manitoba	0.33	10	0.30	0.62	0.71
			50	0.32	0.42	0.59
	Low alkali to special order		1	0.27	0.40	0.53
ST. MARYS	St. Marys, Ontario	0.70	10	0.48	0.99	1.13
			50	0.42	0.89	1.01
LAFARGE CANADA	Woodstock, Ontario	0.50	10/20	0.12	0.47	0.43
			50	0.10	0.43	0.38
			2	0.12	0.46	0.42
			1	0.09	0.53	0.44
ST. LAWRENCE	Mississauga, Ontario	1.60	10	0.21	1.18	0.99
			1	as above approx.		
			30	as above approx.		
	Type 2 to N.Y.D.O.T. specifications		2	no data		
ST. MARYS	Bowmanville, Ontario	0.60	10	0.27	1.40	0.99
	Type 2 to N.Y.D.O.T. specifications		2	0.24	0.55	0.60
LAFARGE CANADA	Bath, Ontario	1.00	10	0.27	0.92	0.88
			30	as above approx.		
LAKE ONTARIO	Picton, Ontario	1.3	10	0.23	1.15	0.98
			30	0.20	1.15	0.94
	Type 2 to N.Y.D.O.T. specifications		2	0.16	0.72	0.63
			5	0.14	0.76	0.65
MIRON	Montreal, Quebec	-	10	0.29	0.45	0.59
	(supply imported cements from Spain & Greece)		10	0.13	0.62	0.54
ST. LAWRENCE	Joliette, Quebec	1.10	10	0.37	0.92	0.98
			1	0.37	0.94	0.99
			20	0.27	0.88	0.85
			50	0.31	0.91	0.91
ST. LAWRENCE	Beauport, Quebec	0.75	10	0.30	0.65	0.73
			30	as above approx.		
			2	0.35	0.40	0.61
CIMENT QUEBEC	St. Basile, Quebec	0.55	10	0.19	1.07	0.89
			20	0.19	1.02	0.86
			30	0.18	1.11	0.91
LAFARGE CANADA	St. Constant, Quebec	0.94	10	0.28	0.91	0.87
			20	0.31	0.93	0.92
			3	0.37	0.95	0.94
	10P contains 8% silica fume		10P	0.20	0.85	0.76
LAFARGE CANADA	Brookfield, Nova Scotia	0.45	10	0.09	1.24	0.90
			50	0.08	0.41	0.35
			3	0.04	1.20	0.83
NORTH STAR CEMENT	Corner Brook, Newfoundland	0.16	10	0.35	1.10	1.07

CANADIAN RESEARCH

In an effort to prevent the universal adoption of restrictions on maximum alkali levels, the Canadian Standards Association Committee on Cement(A5) formed a subcommittee in 1980 to look for and recommend the adoption of rapid test procedures to evaluate concrete aggregates for A.A.R. At present, many of the current procedures require several months or years of testing, with the result that the structure is usually under construction or even completed by the time the aggregate testing is finished. The sub-committee has a membership of about thirty people. Meetings are held at least twice yearly, one of which is combined with a field trip to review interesting cases of A.A.R. A secondary purpose of the committee is to provide a means of communication for those people in Canada involved in work on A.A.R.

A number of authorities are sponsoring research into A.A.R. The Canadian government, through CANMET, is funding investigations by consultants of the incidence of A.A.R. in many of the provinces and funding investigations of Canadian pozzolans. This work is hampered, to some extent, by the lack of reliable, rapid test procedures. The Canadian government also funds work by university researchers through NSERC operating grants. There has, as well, been federal funding for individual projects (Chen and Grattan-Bellew). Some provinces also fund A.A.R. research. This work is either performed or funded by provincial authorities such as electrical utilities or transportation departments. Over the past five years, the total amount of money spent in Canada on investigations and testing of A.A.R. probably has not exceeded an average of about $1m yearly. The amount currently spent on research is small compared to the direct and indirect costs of damage caused by A.A.R. However, this is not as important as the presence of experienced, dedicated researchers, which we are fortunate to have in Canada.

EVALUATION OF AGGREGATES

The first step recommended by CSA in the evaluation of the suitability of concrete aggregates is a thorough petrographic examination and investigation of prior field performance. This will determine what tests, if any, are needed. Failure to properly classify the aggregate or to recognize minor deleterious components will upset the remainder of the testing program. Petrographic examination should, if possible, be based on a knowledge of the prior field performance of similar aggregate. A simple classification of the rock types and minerals present is rarely satisfactory. In Canada, the acquisition of field performance is often impossible due to construction in remote and undeveloped areas.

Many of the tests used in Canada have been found to suffer from such problems as lack of correlation with field performance or poor multilaboratory precision. The concrete prism expansion test is being subjected to a multilaboratory study to find less variable storage conditions than the conventional humid curing room. The mortar bar expansion test has been found to be susceptible to alkali leaching caused by the presence of wicks in the storage container (Rogers and Hooton, 1989).

The CSA sub-committee has established a draft procedure for the accelerated mortar bar test (1 molar NaOH @ 80°C for 14 days) of Oberholster and Davies (1986). This test appears reasonably precise and capable of recognizing the majority of reactive aggregates found in Canada. A multilaboratory study has been conducted to finalize some details and investigate precision. It is likely that only aggregate giving more than 0.20% expansion at fourteen days will be classed as potentially reactive (Hooton, 1990).

The quick chemical test (ASTM C 289) is generally unsatisfactory with alkali-silica reactive carbonate aggregates because much of the alkali-soluble silica released is re-precipitated as calcium silicate hydrates. Researchers in Quebec (Berard and Roux, 1986, Fournier et al, 1987) have had some success with modifying the test so that it is done on the insoluble residue of quarried carbonate rocks. This test is described in a draft CSA document and has been investigated in a multilaboratory study (Fournier, Berube, 1990).

A rapid procedure of expansion testing of concrete cores proposed by Scott and Duggan (1987) is being studied by researchers at the Universities of Calgary and Manitoba to determine the mechanism of expansion. This procedure has similarities to the Conrow test (ASTM C 342) and, at present, does not appear to be satisfactory for separating reactive from non-reactive aggregate (Jones et al, 1990).

To assist in calibration of existing and new procedures, the Ontario Ministry of Transportation has established three 110-tonne stockpiles of known reactive aggregates. Aggregates from these stockpiles are available free of charge for calibrating test procedures or research into A.A.R.

CORRECTIVE MEASURES

The use of low alkali cement is not a universally satisfactory solution because of its lack of availability in eastern Canada. In Alberta, an engineer recently proposed the use of a maximum cement alkali content of 0.3% for dam construction. Such a cement is not available in Canada. Such caution is, nevertheless, understandable, given the high cost of remedial work on dams.

Reducing cement contents and, hence, alkali levels is often not practical because of the severe Canadian climate which generally requires low w/c ratio, air-entrained concrete for all exposed work. In practical terms, this calls for minimum cement contents in the order of 325-450 kg/m^3 if durability is to be ensured.

The use of blast furnace slag cement has generally not been adopted to control A.A.R., despite its apparent efficiency at high levels of substitution (>50%). Laboratory salt-scaling tests (ASTM C 672) and limited field performance has shown that, at 50% substitution levels, the scaling loss from the surface may become unacceptable. For this reason, the Ontario Ministry of Transportation only permits a maximum of 25% substitution.

Other pozzolans such as fly ash and silica fume are used in Canada, but rarely for the purpose of controlling A.A.R. A notable exception was the successful use of 20% fly ash by Ontario Hydro on the Lower Notch Dam to prevent an alkali-silicate reaction with an argillite (Sturrup et al, 1983). Pozzolans are more commonly used for their other advantages such as reduced cost, increased strength-producing properties, or reduced heat of hydration.

The most commonly used control measure is the beneficiation or selective quarrying of aggregate. A technique used in areas of Ontario that contain significant amounts of chert in glacial drift is to selectively crush oversize gravel (>40 mm) which normally contains much less chert. Other techniques used are heavy media separation to remove shale and chert or jigging, which has been used in the past but with limited success. Reserving a specific level or bench of superior quality in carbonate bedrock quarries is a common practice. The underlying or overlying beds may be alkali-reactive, but careful, conscientious extraction and stockpiling can ensure an adequate supply of non-reactive rock.

THE NEXT TEN YEARS

New cases of A.A.R. will be discovered in Canada. A group of reliable, rapid test procedures will become available for each type of reaction. Each of these tests will have known multilaboratory precision and will be calibrated with known reactive aggregates. Corrective measures will generally only be used where non-reactive aggregates are more expensive. There will be more frequent specification of low or reduced alkali cements, and a more widespread adoption of lists of pre-qualified, non-reactive aggregate which only Ontario uses at present.

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REFERENCES

BERARD, J., ROUX, R.; La Viabilite des Betons due Quebec: le role des granulats; Canadian Journal of Civil Engineering, 13, 1, 12-24, 1986.

CANADIAN STANDARDS ASSOCIATION; Appendix B - Alkali-Aggregate Reaction; Canadian Standards Association, Rexdale, Ontario, Canada. Supplement No. 2 to Standards CAN3-A23.1-M77 and CAN3-A23.2-M77, October, 1986, 37p.

CHEN, H. and GRATTAN-BELLEW, P.E.; Effect of Cement Composition on Expansion of Mortar Bars Due to Alkali-Silica Reaction; Proceedings of the 8th International Conference on Alkali-Aggregate Reaction in Concrete, Kyoto, Japan, pp 635-640, 1989.

FOURNIER, B., BERUBE, M.A., and VEZINA, D.; Investigation of the Alkali-Reactivity Potential of Limestone Aggregates from the Quebec City Area (Canada); Proceedings of the 7th International Conference on Alkali-Aggregate Reaction in Concrete, Ottawa, Canada, 1986; Noyes Publications, Park Ridge, N.J., U.S.A.; 23-29, 1987.

FOURNIER, B. and BERUBE, M.A.; Evaluation of a Modified Chemical Method to Determine the Alkali-Reactivity Potential of Siliceous Carbonate Aggregates; Proceedings Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Rectivity; Ministry of Transportation, Ontario, Engineering Materials Report 92, pp 118-135, 1990.

GRATTAN-BELLEW, P.E., SEREDA, P.J., and DOLAR-MANTUANI, L.; The Aggregate Shortage and High Alkali Cement in a Changing Energy Situation; Canadian Journal of Civil Engineering, 5, 250-261, 1978.

HOOTON, R.D.; Interlaboratory Study of the NBRI Rapid Test Method and CSA Standardization Status; Proceedings Canadian Developments in Testing Concrete Aggregate for Alkali-Aggregate Reactivity; Ministry of Transportation, Ontario, Engineering Materials Report 92, pp 225-240, 1990.

JONES, T., GRABOWSKI, E., QUINN, T., GILLOTT, J.E., DUGGAN, R., and SCOTT, F.; Mechanism of Expansion in Duggan Test for Alkali-Aggregate Reaction; Proceedings Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; Ministry of Transportation, Ontario, Engineering Materials Report 92, pp 70-82, 1990.

OBERHOLSTER, R.E. and DAVIES, G.; An Accelerated Method for Testing the Potential Alkali-Reactivity of Siliceous Aggregates: Cement and Concrete Research, 16, 181-189, 1986.

ROGERS, C.A.; Testing Canadian Aggregates for Alkali-Reactivity; Proceedings of the 7th International Conference on Alkali-Aggregate Reaction in Concrete, Ottawa, Ontario, Canada, 1986; Noyes Publications, Park Ridge, N.J., U.S.A.; 259-263, 1987.

ROGERS, C.A. and WORTON, S.; Alkali-Aggregate Reactions in Canada - A Bibliography; Ministry of Transportation, Ontario, Engineering Materials Office, Report EM-83, 3rd Edition, February, 1988, 26p.

ROGERS, C.A. and HOOTON, R.D; Alkali Leaching in Alkali-Aggregate Reaction Expansion Tests; Proceedings of the 8th International Conference on Alkali-Aggregate Reaction in Concrete, Kyoto, Japan, pp 327-332, 1989.

SCOTT, J.F. and DUGGAN, R.; Potential New Test for Alkali-Aggregate Reactivity; Proceedings of the 7th International Conference on Alkali-Aggregate Reaction in Concrete, Ottawa, Ontario, Canada, 1986; Noyes Publications, Park Ridge, N.J., U.S.A.; 319-323, 1987.

STURRUP, V.R., HOOTON, R.D., and CLENDENNING, T.G.; Durability of Fly Ash Concrete; American Concrete Institute, Detroit, U.S.A., SP-79, Vol.1, 71-86, 1983.

SWENSON, E.G.; Cement-Aggregate Reaction in Concrete of a Canadian Bridge; ASTM Proceedings, 57, 1043-1056, 1957.

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EVALUATION OF RAPID TEST METHODS FOR DETECTING ALKALI-REACTIVE AGGREGATES

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ABSTRACT

Using 12 aggregates, mostly from Ontario, with known petrographic characteristics and field performances, a series of rapid test methods was evaluated along with standard ASTM C 227 mortar bar tests.

The rapid test methods involving mortar bars included those of Oberholster and Davies (14 days in NaOH at 80°C), Chatterji (8 to 20 weeks in NaCl at 50°C), Duncan (100% rh at 64°C), Nishibayashi (added NaOH in bars, steamed at 125°C for 4 hours), and Tang (steam cured, then autoclaved in KOH at 150°C for 6 hours).

While some tests are still in progress, the NBRI 14-day test published by Oberholster and Davies appears to be the most promising in terms of distinguishing between non-reactive, marginal and reactive aggregates. However, the expansion limits proposed by Oberholster and Davies may only be suitable for distinguishing non-reactive and very reactive aggregates. Other testing may still be required to distinguish slowly expansive aggregates from petrographically marginal aggregates with good field performance. As well, it was found that washing of the aggregate normally done in ASTM C 227 to remove dust, may not be required with this test.

While some of the other mortar bar methods may have been unfavourably influenced by the authors' decision to modify them by using ASTM standard mortar bars (25 mm by 25 mm cross-section) and C 227 aggregate gradations, many of the methods could not distinguish all of the reactive aggregates.

Based on these and other findings, the CSA Standards Committee is presently investigating the possible adoption of the 14-day NBRI procedure.

1. INTRODUCTION

There is an urgent need to develop and standardize the use of reliable rapid test methods for detecting deleteriously alkali-reactive aggregates. The widely used ASTM C 227 mortar bar

method involves waiting at least 6 months and is not reliable in a reasonable time frame for detecting slowly-reactive aggregates. Numerous rapid methods have been proposed but there was a need to evaluate the more promising ones with respect to existing standard methods and known field performance within the Province of Ontario.

2. EXPERIMENTAL

2.1 Materials

A single, high alkali (Na_2O = 0.39%, K_2O = 1.18%, Na_2O = 1.17%) portland cement was used. The 12 aggregates, mostly from Ontario, are described by their petrographic and field performance in Table 1. Included are known non-reactive, deleteriously reactive, and petrographically marginal aggregates.

2.2 Test Methods

2.2.1 ASTM C 227 The bars were stored upright at 100% rh and 38°C in wicked, rectangular, stainless steel containers capable of holding 18 bars. Inside the containers, three of the six bars in each set were placed in heat-sealed, polyethylene bags with approximately 10 ml of water. Previous testing had demonstrated that the bars in the bags would expand more rapidly if the aggregates were reactive. ASTM C 33 suggests expansion limits of 0.05% at 3 months and 0.10% at 6 months but many agencies including Ontario Hydro impose more stringent limits of 0.05% at 6 months and 0.10% at 12 months. However, with late-expanding, alkali-silicate reactive aggregate [1], 0.1% may not be exceeded for 18 to 24 months.

2.2.2 NBRI Method This method described by Oberholster and Davies [2,3] subjects ASTM C 227 mortar bars to a 1N NaOH solution at 80°C for 14 days. The bars are measured hot. Oberholster and Davies have suggested expansion limits of 0.10% for innocuous aggregates, 0.10 to 0.25% for slowly expansive aggregates, and greater than 0.25% for rapidly expansive aggregates [3].

2.2.3 Duncan Method In this method, the expansion of four ASTM C 227 bars was accelerated in an 100% rh, 64°C environment [4]. The 4 bars were sealed in polyethylene bags and then placed horizontally above water in a sealed plastic container. A limit of 0.05% at 16 weeks was suggested [4]. Four bars were cast in each set.

2.2.4 Danish Salt Method ASTM mortar bars were cast (instead of ISO mortar bars) and there were subjected to a saturated NaCl solution at 50°C while three more were subjected to deionized water at 50°C [5]. Deleterious expansions (> 0.10%) in the salt solution, corrected for the expansions in water were reported within 8 weeks for some reactive aggregates and in 20 weeks for others [6].

2.2.5 Japanese Rapid Test In this method, proposed by Nishibayashi et al [7], mortar bars are made with added NaOH to raise the Na_2O eq to 3.0%, then, at 24 hours they are subjected to saturated steam at 125°C (0.15 MPa pressure) for 4 h. In this study, the test was modified using four ASTM C 227 mortar bars and the Na_2O eq. was raised to 4.0% based on preliminary tests with one of the reactive aggregates.

2.2.6 Chinese Autoclave Test Mortar bars are cured at 125°C in steam prior to autoclaving in a 10% KOH solution for 6h at 150°C [8]. Since it was decided to modify all tests to use ASTM

25 mm cross-section mortar bars and ASTM proportions (graded aggregate to cement ratio of 2.25 to 1), this modified method was not successful (ie. None of the aggregates expanded to 0.10%) and it was abandoned. However, with the originally proposed mix proportions and bars, it was found to work well elsewhere with Canadian aggregates [9].

3. RESULTS

The expansions of mortar bars in the various tests are summarized in Tables 2 and 3.

In the ASTM C 227 test (Table 2), only the bars that were sealed in bags expanded at all. The lack of expansion in the wicked containers is consistent with the problem of alkali leaching [10]. For the bagged bars, only the Spratt aggregate (#12) exceeded 0.10% at 12 months. However, several of the other reactive aggregates would be classified as the late-expansive, alkali-silicate type [1] and would be only expected to exceed 0.10% at later ages (Aggregates #8, 10, 11). Bars made with these three aggregates are the only others to have expanded more than 0.05% at 12 months and are approaching 0.10% at 18 months as shown in Table 3.

Of the rapidly methods, NBRI method (Table 2) gave the best results with respect to known field performance. It classified three aggregates as rapidly reactive (> 0.25% at 14 days: #10, 11, 12), five as slowly reactive, marginal or unclear (0.10 to 0.25% at 14 days: #5, 6, 7, 8, 9) and four as innocuous (< 0.10% at 14 days: # 1, 2, 3, 4). Of the unclear NBRI test results, from field performance, #5 and #7 are marginal (poor petrographically but good field performance) and #6, #8 and #9 would be considered slowly reactive.

As well, for the 3 rapidly reactive aggregates (#10, 11, 12), both the 14 day NBRI expansions and the 12 or 18 month ASTM C 227 expansions would rank them in the same order. From the 28 and 56 day expansions included in Table 2, it can be observed that all of the aggregates continue to expand in the hot NaOH solution but the relative ranking of the aggregates remains the same at all ages.

The Duncan test expansion data is given in Table 3 for periods of 1.5, 3 and 6 months. The results are not very encouraging and it appears that sealing ASTM C 227 bars in bags at 38°C is more effective in accelerating reactive expansions than raising the storage temperature to 64°C. However, both reactive aggregates #10 and #12 did expand >0.05% at 16 weeks.

The Danish salt test (Table 3) did not result in any expansions exceeding 0.10% and when the expansions of companion bars in water is subtracted from the expansions in salt solution, the corrected expansions became negligible.

Even with the equivalent alkali content raised to 4.0%, the modified Japanese Rapid method (Table 3) only gave an expansion exceeding 0.10% for the Spratt aggregate (#12). It appears that the original aggregate to cement ratio or the aggregate gradation proposed by Nishibayashi [7] is critical to the success of this method and should not have been altered. The same is likely true for the Chinese Autoclave test.

4. ADDITIONAL TESTS

Based on the success of the NBRI test method in detecting reactive aggregates, it was decided to conduct further tests with some of the aggregates to study the influence of: a) using a fixed water to cement ratio (w/c) of 0.50 rather than varying w/c to obtain a constant flow, and b)

not washing the dust off of the crushed aggregate as required in ASTM C 227. The dust reacts pozzolanically and reduces expansion in the ASTM C 227 test but it was thought that the excess NaOH in the NBRI test would be sufficient to continue expansion even if the dust reacted. Using a fixed w/c and not having to wash the aggregate fractions would simplify the test procedure.

The results are given in Table 4. It can be observed that higher expansions were obtained in some cases when a fixed w/c was used. The lack of washing of the aggregate fraction also did not reduce expansions significantly and in the case of the Spratt aggregate, expansion was increased.

5. CONCLUSIONS AND RECOMMENDATIONS

Based on these results, ASTM C 227 takes more than 18 months to indicate potentially deleterious, late-expanding alkali-silicate aggregates (ie: to obtain > 0.10% expansion).

Sealing the C 227 bars in polyethylene bags accelerates the expansion of reactive aggregates, which has been found elsewhere to be due to reduced alkali leaching.

Of the five rapid mortar bar test methods evaluated, only the NBRI 14 day test method, developed by Oberholster and Davies, was able to distinguish all of the reactive aggregates. The failure of the other rapid test methods may have been due to modifications made to them or the different types of reactive aggregates used in this study.

While test limits need to be developed for the NBRI test method based on a much larger series of tests, as an interim measure it is suggested that the limits suggested by Oberholster and Davies [3] are appropriate. ie. If less than 0.10% expansion is observed after 14 days in NaOH, then the aggregate is innocuous. Also, if more than 0.25% expansion is obtained, then the aggregate is likely reactive. However, expansion results between 0.10 and 0.25% are less clear. It appears that aggregates with good fold performance but which are petrographically marginal, expanded up to 0.175% at 14 days (#5 and #7). As well, some slowly reactive aggregates also expand approximately the same (#6 and #8). Therefore, for expansions between 0.10% and 0.25%, further testing and evaluation is required. In all cases, it is cautioned that petrographic examination is required to ensure that high expansions are in fact due to alkali-aggregate reactivity.

Based on this work and unpublished work by other Canadian researchers, draft CSA and ASTM [11] test procedures based on the NBRI method have been written by the authors and interlaboratory test programs are being undertaken to establish precision data.

ACKNOWLEDGEMENT

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REFERENCES

[1] Alkali-aggregate Reaction, CSA CAN3-A23.1-M77 Appendix B, Canadian Standards Association Supplement No. 2, 37pp, 1986.

[2] Oberholster, R.E. and Davies, G., An Accelerated Method for Testing the Potential Alkali Reactivity of Siliceous Aggregates, Cement & Concrete Research, 16, 2, 181, 1986.

[3] Davies, G. and Oberholster, R.E., An Interlaboratory Test Programme on the NBRI Accelerated Test to Determine the Alkali-reactivity of Aggregates, National Building Research Institute, CSIRO Special Report BOU 92–1987, Pretoria, RSA, 16pp, 1987.

[4] Duncan, M.A.G., Swenson, E.G., Gillott, J.E., and Foran, M.R., Alkali-Aggregate Reaction in Nova Scotia I. Summary of a Five Year Study, Cement & Concrete Research, 3, 1, 55, 1973.

[5] Chatterji, S., An Accelerated Method for the Detection of Alkali-Aggregate Reactivities of Aggregates, Cement & Concrete Research, 8, 5, 647, 1978.

[6] Thaulow, N. and Olafsson, H., Alkali-Silica Reactivity of Sands, Comparison of Various Test Methods, Nordtest Project, Proceedings, 6th International Conference on Alkalis in Concrete, Research and Practice, Copenhagen, 359, 1983.

[7] Nishibayashi, S., Yamura, K. and Matsushita, H., A Rapid Method of Determining the Alkali-aggregate Reaction in Concrete by Autoclave, Proceedings, 7th International Conference on Concrete Alkali-Aggregate Reactions, Ottawa, 299, 1986.

[8] Tang, Ming-shu, Han, Su-fen, and Zhen Shi-hua, A Rapid Method for Identification of Alkali Reactivity of Aggregate, Cement & Concrete Research, 13, 3, 417, 1983.

[9] Grattan-Bellew, P.E., Personal Communcation, National Research Council of Canada, Feb. 1989.

[10] Rogers, C.A. and Hooton, R.D., Leaching of Alkalies in Alkali-Aggregate Reaction Testing, Proceedings, 8th International Conference on Alkali-Aggregate Reaction, Kyoto, 327, 1989.

[11] ASTM C-9 Proposal P 214 Proposed Test Method for Accelerated Detection of Potentially Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction, 4pp, 1990.

[12] Hooton, R.D. and Rogers, C.A., Evaluation of Rapid Test Methods for Detecting Alkali-Reactive Aggregates, Proceedings, 8th International Conference on Alkali-Aggregate Reaction, Kyoto, 439, 1989.

TABLE 1

Aggregate Description and Field Performance

Aggregate Name	Description	Reactive Component	Field Performance
Nelson stone	quarried dolostone	none	Excellent - > 30 years
Ottawa sand (Illinois)	pure quartz sand	none	None
Fleming stone	quarried granite	strained quartz	None[4]
Guelph sand	carbonate sand	0.2% chert	Excellent - > 30 years
Paris sand	carbonate sand	0.3% chert	Excellent - > 30 years
Neyes sand	siliceous sand	2.5% chert	Reactive[2]
Paris stone	carbonate gravel	1.6% chert	Excellent - > 30 years
Grant stone	siliceous gravel	27% argillite, greywacke, sandstone	Reactive[1]
Natural sand (Michigan)	siliceous sand	7.4% chert	Reactive[1]
Sudbury stone	siliceous gravel	75% argillite, greywacke, sandstone	Reactive[1]
Lower Notch stone	quarried meta-sediments	98% argillite, greywacke, sandstone	Reactive[3]
Spratt stone	quarried limestone	< 5% chalcedony	Reactive[1]

Footnotes:1. Alkali reaction identified in many structures.
2. Alkali reaction identified in part of one 30-year old structure.
3. Alkali reaction identified in small part of one 20-year old dam (but the use of fly ash has prevented reaction in most of this structure).
4. Petrographically similar to alkali-silica reactive granite in 70-year structure.

TABLE 2

ASTM C 227 and NBRI Test Expansions (%)

#	AGGREGATE NAME	ASTM C 227						NBRI METHOD			
		MTO CONTAINER			BAGGED						
		6m	12m	18m	6m	12m	18m	12d	14d	28d	56d
1	Nelson	.019	.034	.036	.021	.036	.040	.009	.012	.020	.039
2	Ottawa	.023	.022	.026	.019	.021	.023	.038	.038	.053	.139
3	Fleming	.005	.021	.019	.021	.039	.040	.038	.046	.086	.205
4	Guelph	.029	.039	.041	.029	.042	.049	.077	.093	.177	.297
5	Paris Sand	.023	.039	.049	.029	.047	.050	.122	.153	.306	.399
6	Neyes	.024	.024	.030	.020	.020	.028	.118	.164	.369	.513
7	Paris	.012	.025	.020	.025	.045	.044	.144	.175	.293	.336
8	Grant	.011	.024	.014	.030	.054	.057	.143	.185	.423	.533
9	Natural	.023	.023	.031	.041	.048	.052	.172	.213	.494	.525
10	Sudbury	.022	.031	.032	.036	.062	.080	.221	.266	.466	.736
11	Lower Notch	.017	.032	.035	.036	.077	.094	.282	.326	.540	.746
12	Spratt	.046	.055	.058	.166	.222	.234	.309	.356	.626	.956

TABLE 3

Duncan, Danish Salt and Japanese Rapid Test Expansions (%)

#	AGGREGATE NAME	DUNCAN TEST			DANISH SALT TEST			JAPANESE RAPID TEST
					in Sat. NaCl		CORRECTED	
		12w	16w	26w	8w	20w	20w	4% NaOH
1	Nelson	.033	.023	.025	.003	.013	.001	.034
2	Ottawa	.027	.028	.019	–	–	–	.033
3	Fleming	.040	.044	.050	.022	.054	.008	.047
4	Guelph	.032	.034	.036	.003	.007	.007	.060
5	Paris Sand	.036	.035	.037	.008	.019	.005	–
6	Neyes	.035	.042	.061	.022	.027	.003	.057
7	Paris	.035	.034	.037	.017	.026	.007	.060
8	Grant	.029	.033	.041	–	–	–	.043
9	Natural	.025	.026	.025	–	–	–	.054
10	Sudbury	.057	.055	.063	.016	.049	.002	.044
11	Lower Notch	–	–	–	.016	.023	.008	.069
12	Spratt	.068	.071	.081	.021	.054	.013	.225

TABLE 4

Influence of Test Variations on NBRI Expansions

#	AGGREGATE NAME	14 DAY EXPANSIONS (%)			W/C FOR CONSTANT FLOW
		CONSTANT FLOW	W/C = 0.50	UNWASHED AGGREGATE CONSTANT FLOW	
5	Paris Sand	.153	.197	.147	.46
7	Paris	.175	.163	.151	.54
10	Sudbury	.266	.255	.275	.53
11	Lower Notch	.326	.403	–	.54
12	Spratts	.356	.375	.441	.54

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

CANADIAN EXPERIENCE WITH THE MORTAR BAR ACCELERATED TEST FOR ALKALI-AGGREGATE REACTIVITY

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ABSTRACT

The expansivity of a series of different types of aggregates was evaluated in the concrete prism test, and in the mortar bar accelerated test. The compositions of the aggregates were determined by petrographic examination. Moderately good correlation was found between expansions in the concrete prism and mortar bar accelerated tests. Good correlations were also found between the composition of the aggregates and expansion in the mortar bar accelerated test.

1. INTRODUCTION

A number of accelerated test methods for alkali-aggregate reactivity have been proposed. Most rely on high temperature, and or pressure, to speed up the expansion of mortar bars.[1]. All the proposed test methods appear to be effective, and the problem facing the construction industry is not so much which method to select, but rather how to establish acceptable expansion limits for evaluating concrete aggregates. Greater expansions are obtained using the accelerated methods than with the traditional mortar bar or concrete prism tests. This is advantageous, as standard test methods, currently in use, underestimate the long term expansion potential of concrete in the field. However, care will have to be exercised to prevent some aggregates from being rejected; these are ones which although reactive with alkalis, would perform satisfactorily in field concretes. This is particularly true for aggregates which will be used in concrete subjected to mild exposure conditions. Separate expansion limits may have to be established, for the evaluation of aggregates, in accelerated tests, subject to various exposure conditions. It may also prove necessary to establish separate expansion limits for the evaluation of different types of rocks, e.g. greywacke, siliceous limestone, silicified volcanics, chert, and sandstone.

The most reliable expansion limits for different types of aggregates are obtained by evaluating those with documented field performance in concrete. However, evaluation of aggregates which have not previously been used in concrete, still poses a problem. This can be overcome by a making petrographic evaluation of the aggregate in conjunction with the accelerated tests. Once the composition of the aggregate has been determined, the expansion obtained can be compared to that from a similar aggregate, with known field performance. The use of petrographic evaluation in conjunction with accelerated testing should prevent incorrect conclusions being drawn from the test results. It is recommended that petrographic examination of the aggregate should be a mandatory part of any accelerated test method, as there will be a strong temptation for those in the construction industry to run accelerated tests blindly, possibly resulting in the rejection of aggregates which would perform satisfactorily in field concrete.

In this paper, the results of the evaluation of a number of aggregates from across Canada in the mortar bar accelerated test [2] (subsequently referred to as the MBAT test) will be discussed. In the MBAT test, length change of the mortar bars is monitored during two weeks of storage in 1 M NaOH @ 80°C. In the analysis of the test results, the aggregates are divided into mineralogically similar groups. The source distribution of the aggregates is shown in figures 1a & 1b.

2. EXPERIMENTAL

2.1 EXPANSION TESTING

2.1.1. MBAT Test

The proposed Canadian standard mortar bar accelerated test procedure (MBAT) was followed. [2] The mortar bars were made according to ASTM C227, [3] except that the mortar was mixed at a constant w/c ratio of 0.50 instead of at constant flow, as specified. This was done to reduce the amount of aggregate required for the test.

2.1.2. Concrete Prism Test

The concrete prism results reported in this manuscript were obtained over a period of about 15 years. During this period the mix design of the concrete has undergone some changes. The basic procedure followed is that specified in CSA A23.2-14A. [4] All the concrete prisms were stored at 38°C. In some instances the cement content of the concrete mix used was 410 kg/m^3 instead of 310 kg specified; this was done to insure that the slowly reacting aggregates such as those from Sudbury expanded. The expansions quoted in this manuscript are the maximum expansions obtained when the expansion curve had flattened out. Due to variations in mix design and to the problem of leaching in the storage containers, the expansions quoted should be regarded as comparative numbers, indicating expansivity, rather than an absolute measure of the expansiveness of the aggregates in concrete.

Figure 1. Location maps for aggregate samples

(a) Map of Canada
(b) Map of Eastern Canada

Locations of aggregates are indicated by black dots, except for those in Alberta, where the area of occurrence of sandstone in shown by a row of hatching.

2.2. Petrographic Evaluation of Aggregates

The composition of ledge rock aggregates was determined by examination of thin sections. In the case of gravel aggregates, in addition to thin section examination, the particles were categorized by visual examination supplemented by simple physical tests. Visual categorization is necessary as the number of particles examined even in large thin sections, (50 x 75 mm) is statistically too small. In some instances, there is some uncertainty in the composition of the gravels as the amount of material available for evaluation was inadequate.

3. RESULTS & DISCUSSION

3.1. Greywackes

Apart from siliceous limestones, greywackes are the most commonly occurring type of reactive aggregate in eastern Canada. Greywackes are found in the Sudbury region of Ontario, in Quebec City, around Fredericton and in southern New Brunswick, and in the Halifax region and elsewhere on the east coast of Nova Scotia. The expansion of mortar bars in the MBAT test and of concrete prisms (CAN3. A23.2-14A) containing a variety of greywackes is shown in figure 2. [3]. For comparison purposes, the expansion of mortar bars made with the reactive Malmsbury greywacke from Cape Town, South Africa is also shown. The Malmsbury aggregate was used in the development of the accelerated mortar bar (MBAT) method. [5]

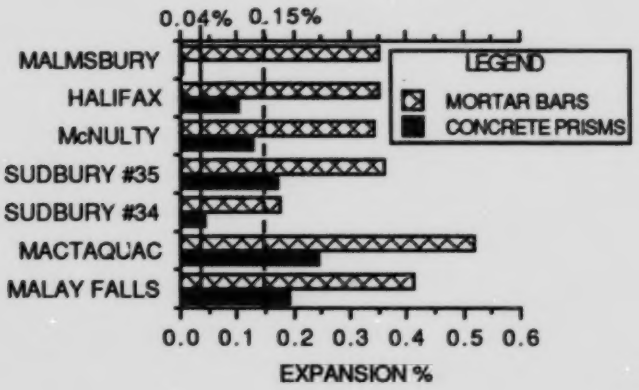


Figure 2. Expansion of greywacke aggregates in concrete prism and MBAT tests. The expansion limit for concrete prisms of 0.04% is shown as a solid line. The 0.15% expansion limit for the MBAT test is shown by a dashed line.

All the greywackes were found to be expansive in both the concrete prism and in the MBAT tests. With the exception of the greywacke from Mactaquac, which gave very large expansions, in both test methods, and of greywacke #34 from Sudbury which gave low expansions, other greywackes gave rise to remarkably uniform expansions, particularly in the MBAT test. The average expansion in the MBAT test is 0.36%.

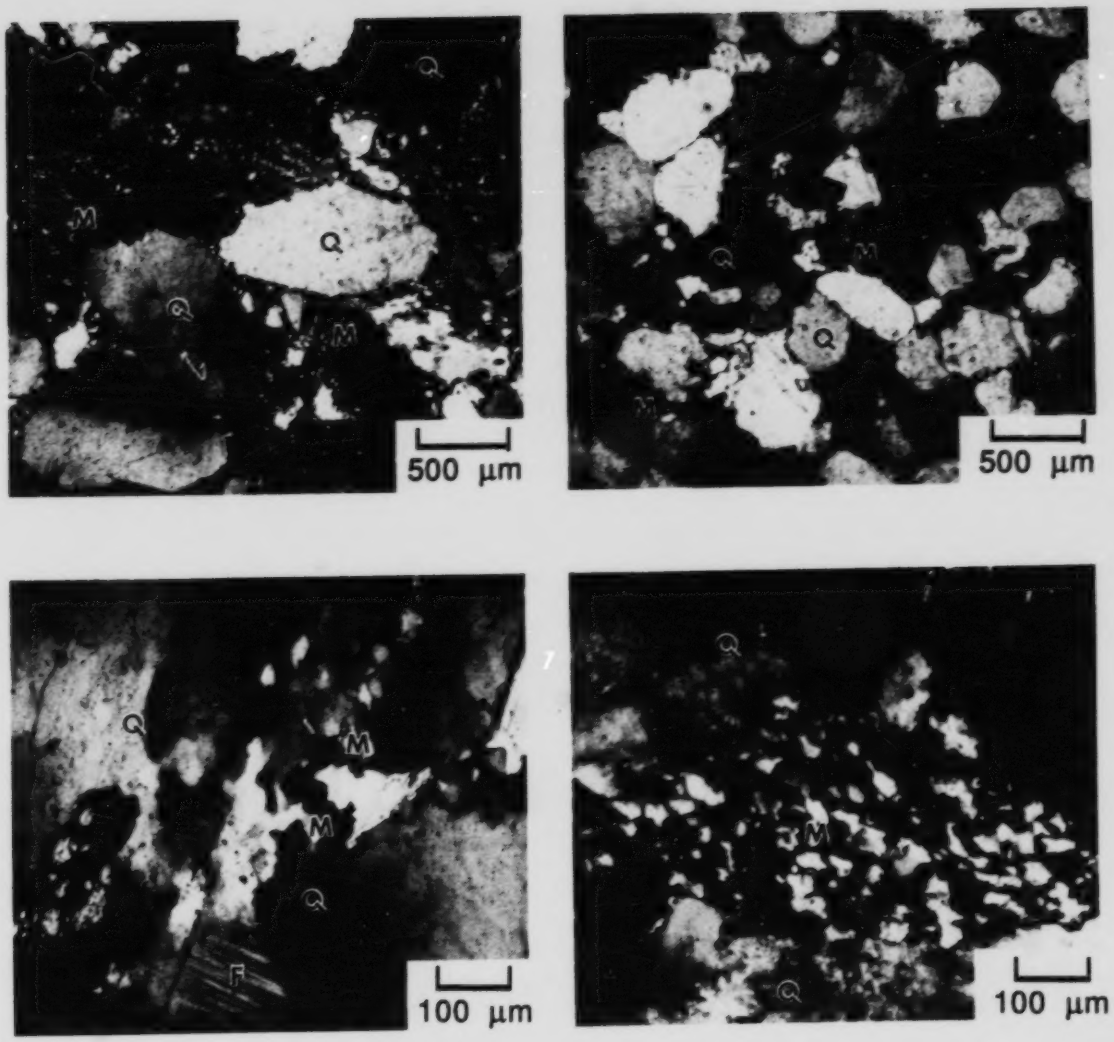


Figure 3. *Optical micrographs, taken between crossed polarizers.*

(a) *Greywacke #35 from Sudbury gravel, showing large areas of interstitial micro-crystalline quartz (M) between the large quartz grains (Q).*

(b) *Greywacke #34 from Sudbury gravel, showing small areas of micro-crystalline quartz between the large quartz grains (Q) which comprise most of the rock.*

(c) *Barre granite showing a few grains of micro-crystalline quarta (M) along grain boundaries. Q-large quartz grains, F- feldspar.*

(d) *Granite from Swift Current showing large areas of micro-crystalline quartz (M) amongst the large quartz grains (Q).*

Micro-crystalline quartz which is equivalent to chert, is probably the reactive component in the greywackes. Chert, consisting of micro-crystalline and crypto-crystalline quartz, similar to that shown filling interstitial space between quartz grains in figure 3a, is extremely expansive in the MBAT test, figure 4.

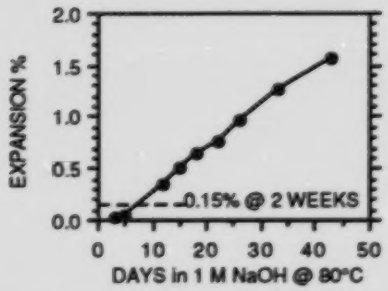


Figure 4. Expansion of mortar bars containing chert from Namibia in the MBAT test. The 0.15% expansion limit is shown by a dashed line.

Petrographic examination of greywackes #34 and #35 from Sudbury showed that #35 contains much more micro-crystalline quartz, figures 3a & 3b. The quantity of micro-crystalline quartz in nine greywackes, including the seven shown in figure 2, was estimated by the point count method in the thin sections. The amount of micro-crystalline quartz is plotted against expansion in the MBAT test in figure 5. The correlation coefficient for the line fitted to the points in figure 5 is 0.819. The Malmsbury greywacke does not fit the line very well; this may be due to variability in the composition of this aggregate. The amount of micro-crystalline quartz in a thin section made from greywacke from Quebec City was determined as 17%. The expected expansion for mortar bars made with this greywacke determined from figure 5 would be 0.23%; the expansion determined in the Laval University laboratory was 0.20% [6]. Concrete prisms made with Quebec City greywacke only expanded by 0.01% at one year, [6] indicating that this aggregate is possibly not deleteriously reactive. All the greywackes shown in figure 5, with the possible exception of Sudbury #34, are known to cause deterioration in concrete in highway structures or dams. The Sudbury gravel, consisting of greywacke, argillite and non-reactive granitic and norite rock caused deterioration of concrete highway structures. [7]

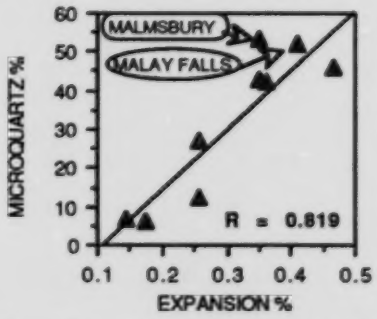


Figure 5. Plot of the percentage of micro-crystalline quartz in greywackes versus the percentage expansion of mortar bars in the MBAT test.

Expansions in the MBAT test of several gravel samples, from pits in the Sudbury region, were in the range of 0.211% to 0.263%; these results indicate that the expansion limit for greywackes in the MBAT test should be around 0.20%. Most greywackes show expansions greater than 0.2%, figure 5.

3.2 Argillites

Argillites are mineralogically related to greywackes and grade into them. They consist of a very fine grained matrix of micro-crystalline quartz and clay in which occur small isolated quartz grains. Only two argillites were evaluated, one the well documented material from Lady Evelyn Lake [6], the other from the Sudbury gravel. In the MBAT test they show the same range of expansions as the greywackes. Both argillites were found to be expansive in both the concrete prism and MBAT tests figure 6.

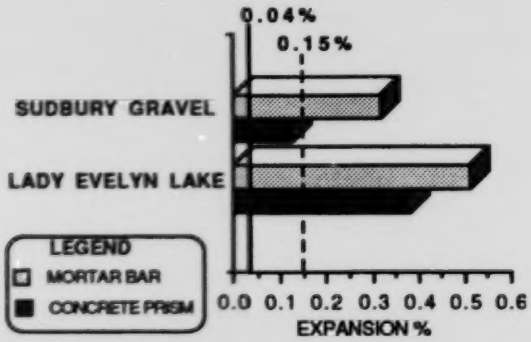


Figure 6. Expansion of mortar bars in the MBAT test, and of concrete prisms, containing argillites. The expansion limits for mortar bars, and concrete prisms, are indicated by solid and dashed lines, respectively.

3.3 Carbonate Rocks

3.3.1. Types of Reactivity

Carbonate rocks may fall into one of two categories of reactive aggregates, those exhibiting alkali-carbonate reactivity, and those exhibiting alkali-silica reactivity.

3.3.2. Alkali-Carbonate Reactivity.

The expansivity of typical alkali-carbonate reactive aggregates such as the Kingston rock sample 78-16 [8] is vastly underestimated by the MBAT test when compared to the expansion in concrete prisms, figure 7, or in field concrete. Crushing the aggregate to the ASTM C227 grading affects its expansivity in the mortar bar test C227, as well as in the MBAT test. It is concluded that the MBAT test is not suitable for evaluating alkali carbonate reactive aggregates.

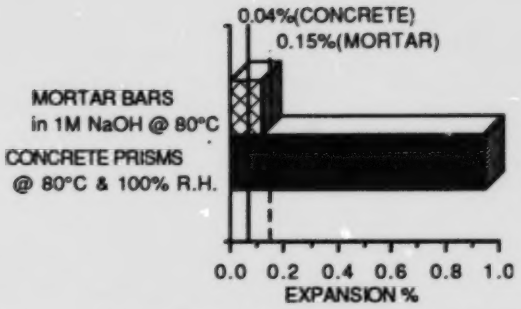


Figure 7. *Expansion of dolomitic limestone from Kingston, in the concrete prism and MBAT tests. The 0.04% expansion limit for concrete prisms, and the 0.15% expansion limit for mortar in the MBAT test, are indicated by solid and dashed lines, respectively.*

3.3.3. Alkali-Silica Reactivity

A large number of siliceous limestone aggregates from Ontario and Quebec have been evaluated in the concrete prism test and MBAT tests. Some of these, for example the well known Spratt limestone, exhibit alkali-silica reactivity.[9]. Well documented, innocuous aggregates, such as the Nelson dolostone, show very little expansion in either test, figure 8. Limestone from Manitoulin Island, which is used in the Windsor area, and from Arnprior were also found to be non expansive in both tests. For reference purposes, expansions of concrete and mortar containing the well documented Spratt limestone [8] from Ottawa are also shown in figure 8.

A sample of limestone from a quarry in the Trois Rivieres region of Quebec was found to be expansive in the concrete prism test when the cement content of the concrete was 420 kg/m^3; it expanded by 0.07% in one year. In the MBAT test, the aggregate would be classified as marginally expansive if a limit of 0.1% is used, but non-expansive if the limit is 0.15%. These results suggest that the aggregate is marginally expansive in concrete. Concrete slabs made with Trois Rivieres aggregate and 420 kg cement/m^3 concrete, exposed in a parking lot at NRC for 5 years, show no evidence of deterioration, despite being heavily salted.

It is concluded from the test results reported above, and from other results obtained by the author, that an expansion limit in the range of 0.1 to 0.15% would be appropriate for the evaluation of the expansivity of potentially alkali-silica reactive carbonate aggregates.

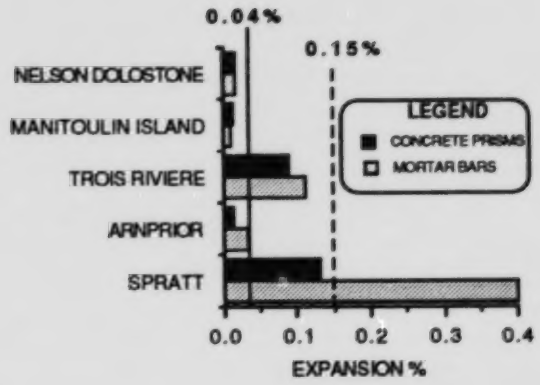


Figure 8. Expansion of mortar bars, in the MBAT test, and of concrete prisms, containing carbonate aggregates. The 0.04% expansion limit for concrete prisms and the 0.15% expansion limit for mortar bars are indicated by solid and dashed lines, respectively.

3.3.4. Carbonate Aggregate in Gravels.

Carbonate aggregates occur in gravels which are used as concrete aggregate in the Winnipeg region of Manitoba. These gravels contain varying proportions of granite, dolostone, limestone and a few percent of chert, figure 9a. The presence of chert makes these aggregates potentially reactive. Evaluation of two gravels in both the MBAT and concrete prism tests shows them to be non-reactive, figure 9b; expansion in both test methods is well below the proposed limit of 0.15%.

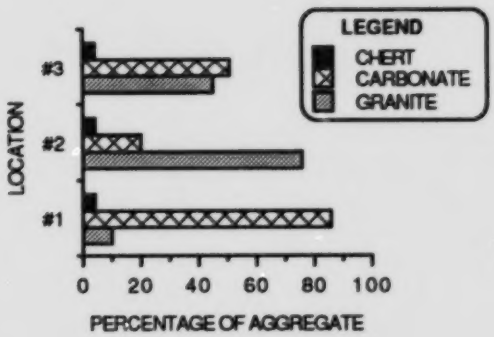


Figure 9 a. Composition of 3 gravels from the Winnipeg area.

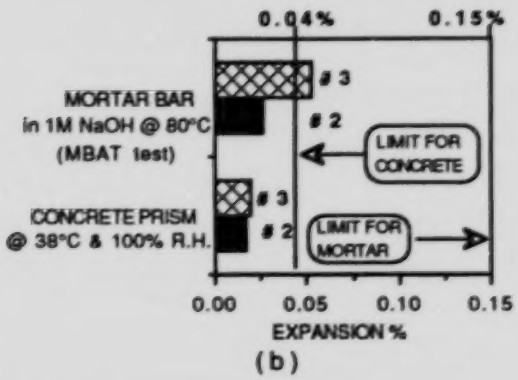


Figure 9 b. Expansion of gravels #2 & #3 from the Winnipeg area, in concrete prism and MBAT tests. Expansion limits for concrete prisms and mortar bars are indicated.

3.4. Plutonic Rocks.

3.4.1. Granites

The only documented case of concrete deterioration, due to alkali-aggregate reaction involving granitic aggregate, in Canada, is the Rosedale viaduct in Toronto [10]. Granitic rock has also been identified as the cause of alkali-aggregate reaction in concrete in Australia [11]. The source of the granite used in the Rosedale viaduct is unknown, but it is similar to the granite found in the Muskoka area of Ontario, figure 1b.

The expansivity of four granites was evaluated in the MBAT test, figure 10. The Barre granite was shown to be marginally expansive in the concrete prism test. Both the Swift Current granite, from Newfoundland and that from Porcupine Mountain, Nova Scotia would be classified as expansive if an expansion limit of 0.1% is used for the MBAT test, but if the limit is taken as 0.15% only the granite from Porcupine Mountain would be considered expansive. The granite from Porcupine Mountain is of variable composition and the sample tested may not be representative, as the aggregate appears to perform satisfactorily in concrete.[12] No reports on the use of other granitic aggregates in concrete were available.

Micro-crystalline quartz is thought to be the reactive component in granites [14]. The percentage of micro-crystalline quartz in the granites is shown along with the expansion of mortar bars in the MBAT test in figure 10. It is evident from figure 10 that there is reasonable correlation between the amount of micro-crystalline quartz in the granite and the expansion of mortar bars. In the Barre granite, micro-crystalline quartz occurs sporadically along grain boundaries figure 3c, but in the granite from Swift Current there are larger areas of micro-crystalline quartz, figure 3d.

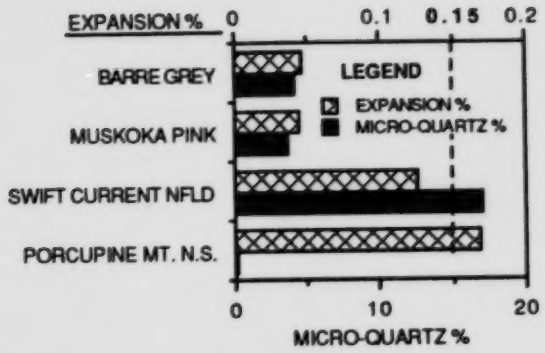


Figure 10. *Percentage of micro-crystalline quartz in granites, and the expansion of mortar bars, containing the same granites in the MBAT test. The 0.15% expansion limit for mortar bars in the MBAT test is indicated by a dashed line.*

3.4.2. Granitic gravel

Gravels containing granite and gneiss are common in the Pre-Cambrian regions of Canada. Several granitic gravels from the Pembroke region of Ontario, figure 1b, were evaluated for potential reactivity using the MBAT test. They showed about the same amount of expansion as the Barre granite and are hence classified as non-expansive. These granitic gravels have a good record of performance in concrete. [14].

3.4.3. Gabbro

A gabbro from Sudbury, Ontario, was evaluated in the MBAT test. The expansion of the mortar bars was 0.051%, indicating that this aggregate should be non-expansive in concrete. Petrographic examination showed that the gabbro consisted of feldspar, amphibole, and accessory minerals. The absence of quartz in this rock is consistent with its low expansion in the MBAT test. The gabbro performs satisfactorily in concrete except that the strength is relatively low due to poor bonding between the aggregate and the cement paste. [14]

3.5. Sandstone

The Potsdam sandstone from the Montreal region is the best documented reactive sandstone in Canada [15]. In this sandstone, about 75% of the sand grains are surrounded by secondary quartz overgrowths. This aggregate does not cause expansion in the MBAT test [6] In Alberta, a sandstone, broadly similar to the Potsdam sandstone, has widespread occurrence, figure 1a. One sample of this rock, the Mount Wilson quartzite, was evaluated in the mortar bar test ASTM C227 ; [8] the results of the mortar bar test indicate that the rock is, at least marginally, slowly expansive. In the Alberta sandstone, only about 45% of the quartz grains have secondary quartz overgrowths, compared to 75% in the Potsdam sandstone. The secondary quartz is thought to be the reactive component in the sandstones. Assuming that the percentage of sand grains surrounded by quartz overgrowths determined for the Potsdam and Alberta sandstones is

representative, then the Potsdam sandstone should be the most reactive. This may not be the case however, as the Alberta sandstone contains zones of micro-crystalline quartz.

The expansions in the MBAT test, of a pure sandstone aggregate, hand picked from the gravels, and two gravels containing sandstone, from Alberta, are shown in figure 11a. For comparison purposes, the expansion of the aggregates in concrete prisms is also shown. It is evident that the sandstone is the most reactive component in the gravels as mortar bars made with the two gravel aggregates show lower expansion than those made with pure sandstone. The composition of a typical gravel is shown in figure 11b. The gravel consists of sandstone, volcanic rocks, limestone and other sediments. Minor cracking, probably due to alkali-aggregate reaction, was observed in some dams and a bridge in southwest Alberta.[16]. Both the Calgary gravel and sandstone gravel #2 caused expansions in the concrete prism test in excess of the 0.04% limit indicating that these aggregates are at least marginally expansive. An expansion limit of 0.15% may be appropriate for the evaluation of the Alberta sandstone gravels.

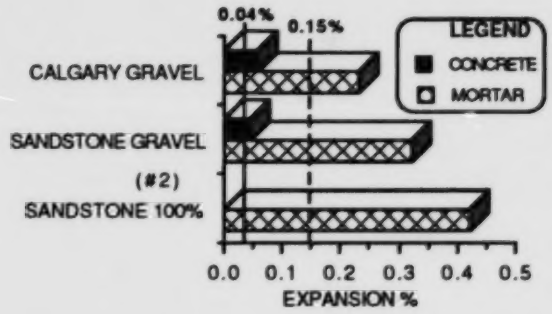


Figure 11a. Expansion of mortar bars in the MBAT test, and of concrete prisms, made with sandstone aggregates. Expansion limits for concrete prisms and mortar bars are indicated by solid and dashed lines, respectively.

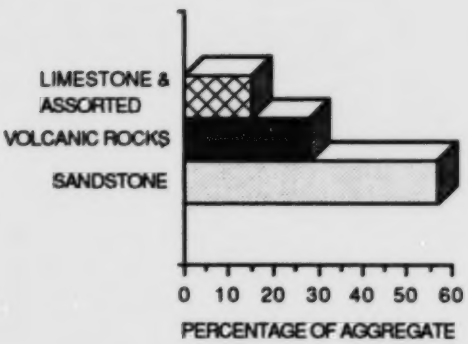


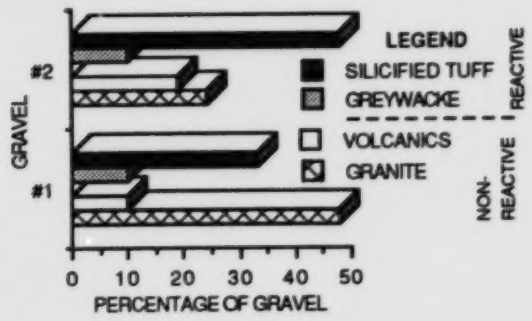
Figure 11 b. Composition of a typical, sandstone bearing gravels from southwest Alberta.

3.6. Silicified Tuff and Devitrified Volcanic Glass

The silicified tuff used as concrete aggregate, in the Sartigan dam, in the Beauce region of Quebec, is the only aggregate of this type known to cause severe deterioration in concrete in Canada. [17] However, petrographically similar rocks occur in the Avalon Peninsula in the Placentia Bay region of Newfoundland, in gravels at Wawa,Ontario, and in some gravels used as concrete aggregate in Vancouver. Examination of thin sections of the tuff from the Beauce showed that it consists of isolated quartz grains of about 0.2 mm grain size, in a matrix of micro-crystalline quartz, feldspar, and sericite. Aggregate from quarry #3, in the Placentia Bay region, which consists of silicified tuff and devitrified volcanic glass, is of similar composition to the tuff from the Beauce.

Many of the gravels in the Placentia Bay region contain silicified tuff and devitrified glass. The composition of typical gravels is shown in figure 12a. About 65% of the gravel particles are composed of potentially reactive tuff, greywacke and chert. The expansion of mortar in the MBAT test, and of concrete prisms, made with a series of aggregates from the Placentia Bay region is shown in figure 12b; expansions of samples made with tuff from the Beauce are shown for comparison.

The Newfoundland aggregates are less expansive than the tuff from the Beauce, in both the concrete prism, and the MBAT test. All the aggregates, with the exception of gravel #2, were shown to be expansive in the concrete prism test; all mortar bars exceeded the 0.15% expansion limit in the MBAT test. In the case of gravel #2 there is no reason to conclude that the MBAT test is giving too much expansion, as it is just as likely that the concrete prism test is giving an incorrect result. Petrographic evaluation shows that gravel #2 from the Placentia Bay region, which contains 60% reactive particles, should be more expansive than gravel #1 with only 45% of reactive particles; this conclusion supports the results given by the MBAT test, figure 12b.



(a)

Figure 12 a. Composition of gravels from the Placentia Bay region of Newfoundland.

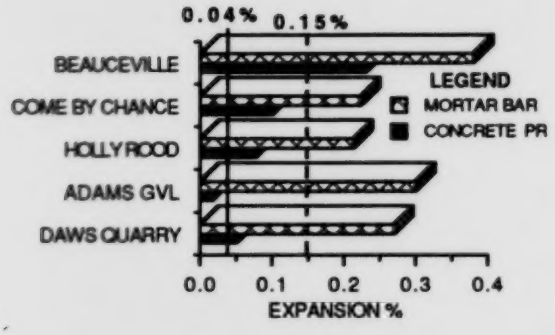


Figure 12 b. Expansion of mortar in the MBAT test, and of concrete prisms, containing silicified volcanic rocks from Newfoundland and from the Beauce region of Quebec. The expansion limits for concrete prisms and mortar bars are indicated by solid and dashed lines respectively.

Silicified volcanic rocks are also found in gravels in Wawa, Ontario and in a gravel used as concrete aggregate in Vancouver. The composition of the gravels is shown in figure 13 a & b. The gravel from Wawa contains about 45% of potentially reactive silicified volcanics and greywacke. The Vancouver gravel contains less than 5% of devitrified glass, an amount, which is probably too small to cause it to be expansive in concrete. The percentage of potentially reactive particles in gravels from Placentia Bay, Wawa, and Vancouver, and the expansivity of mortar bars, containing them, in the MBAT test are shown in figure 13c. It is evident from figure 13c, that the expansivity of the gravels is approximately proportional to their content of potentially reactive particles.

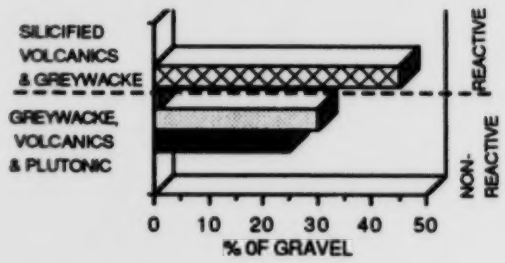
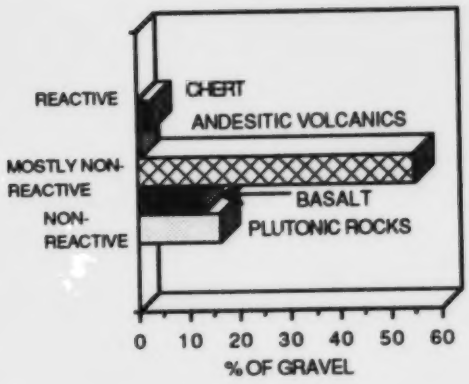
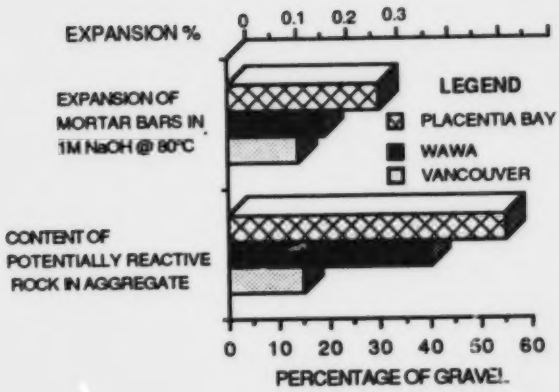


Figure 13a. Composition of gravel from Wawa, Ontario.



(b)



(c)

Figure 13.
(b) Composition of a gravel used in Vancouver
(c) Content of potentially reactive material in the three gravels, and the expansions of mortar bars made with them, in the MBAT test.

Cracking due to alkali-aggregate reactivity was observed in concrete structures made with gravel aggregates, on highways in the Placentia region of Newfoundland, confirming the reactivity of these gravels. An expansion limit of 0.15% in the MBAT test is suggested for aggregates consisting of silicified tuff, or gravels containing a high percentage of it. On the basis of an expansion limit of 0.15%, the Placentia Bay and Wawa gravels would be classified as expansive; the gravel from Vancouver non-expansive, figure 13c. If an expansion limit of 0.1% is used, the gravel from Vancouver would be classified as slowly expansive. It is not possible to draw any conclusion concerning the field performance of the Vancouver gravel, as it has always been used with low alkali cement.

4. CONCLUSIONS

The results of this preliminary survey of Canadian aggregates using the MBAT test indicate that this test would be quite satisfactory as an aggregate screening test, except for alkali-carbonate reactive aggregates, for which it is not appropriate. However, pending further evaluation of the MBAT method, test results indicating that an aggregate is expansive should be checked by running a concrete prism test, preferably using a mix containing 410 kg of cement instead of the 310 kg which is specified in A23.2-14A. Tests, by the author, indicate that there is better correlation between the results of concrete prism tests and field performance, when a 410 kg mix is used.

Despite the wide range of rock types occurring in Canadian aggregates, micro-crystalline quartz is probably the reactive component in all of them, except alkali-carbonate reactive dolomitic limestones. An expansion limit, in the MBAT test, of 0.15 % at two weeks is proposed, for all types of aggregates with the possible exception of siliceous limestones such as the Spratt for which a limit of 0.1% may be needed, particularly for concrete that will be subject to severe exposure conditions. In siliceous limestones, reactive silica occurs as fine divided particles with a grain size in the range of 5 μm to 50 μm which may be more reactive than the micro-crystalline quartz occurring in greywackes. It was suggested that an expansion limit of 0.20% would be appropriate for greywackes, but in the interest of uniformity in the evaluation of aggregates, it may be preferable to err on the safe side, and use the 0.15% expansion limit. The proposed 0.15% expansion limit applies to aggregates for concrete made with high alkali cements such as those commonly used in eastern Canada which have a mean alkali content of 0.95% with a standard deviation of ±0.1%. A higher expansion limit would be appropriate for concretes, made with cements with lower alkali contents, which would not be subject to de-icing salt application.

The principal potential problem with the MBAT test is that some aggregates which may perform satisfactorily in concrete, subject to relatively mild exposure conditions, may be classified as being deleteriously expansive. This danger can be minimized by carrying out a petrographic examination of the aggregate, to determine if it contains sufficient micro-crystalline quartz, to account for the observed expansion in the MBAT test. It is strongly recommended that petrographic examination be made a mandatory part of the mortar bar accelerated test.

5. ACKNOWLEDGMENT

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6. REFERENCES

[1] Grattan-Bellew, P.E., Test Methods and Criteria for Evaluating the Potential Reactivity of Aggregates, Proc. 8th International Conference on Alkali-Aggregate Reaction, Kyoto July 1989. Editor, K. Okada, S. Nishibayashi, and M. Kawamura. The Society of Materials Science of Japan Kyoto 606, Japan.

[2] Proposed Test Procedure for Assessing Likely Alkali-Reactivity of Concrete Aggregates, Cement-Aggregate Reactivity Sub-Committee of CSA-A5, May 1, 1989. Available from C.A. Rogers, Ministry of Transportation, Downsview, Ontario, Canada.

[3] ASTM C227-87 Standard Test Method for Potential Alkali-Reactivity of Cement-Aggregate Combinations (Mortar Bar Method), 1988 Annual Book of ASTM Standards, 4.02, Concrete and Aggregates, ASTM 1916 Race Street, Philadelphia, U.S.A., 121-125.

[4] CAN3-A23.2-14A, Supplement No.2-1986 to CSA Standards CAN3-A23.1-M77, Concrete Materials and Methods of Concrete Construction and CAN3-A23.2-M77, Methods of Test for Concrete. Canadian Standards Association, 178 Rexdale Boulevard, Rexdale, Ontario, Canada, M9W 1R3.

[5] Davies, G. & Oberholster, R.E., An Interlaboratory Test Program on the NBRI Accelerated Test to Determine the Alkali Reactivity of Aggregates, RILEM Document TC-106, AAR (Alkali-Aggregate Reaction) Accelerated Tests, Appendix 2.

[6] Bérubé, M.A., Laval University, Ste-Foy, Quebec. Personal communication.

[7] Magni, E.R., & Rogers, C.A., The Influence of the Alkali-Silicate Reaction on Structures in the Vicinity of Sudbury, Ontario, Ontario Ministry of Transportation, Engineering Materials Office, Report EM-81.

[8] Grattan-Bellew, P.E., & Gillott, J.E., Three Decades of Studying the Alkali Reactivity of Canadian Aggregates, Concrete Durability, Katherine & Bryant Mather International Conference, Ed. John M. Scanlon, ACI SP 100-70, American Concrete Institute, Detroit, U.S.A., Vol.2, 1365-1384, 1987.

[9] Rogers, C.A., General Information on Standard Alkali-Reactive Aggregates from Ontario, Canada, Ontario Ministry of Transportation, Engineering Materials Office.

[10] Rogers, C.A., Alkali Aggregate Reactions, Concrete Aggregate Testing & Problem Aggregates in Ontario, A Review, Ontario Ministry of Transportation, Engineering Materials Office, Report EM-31.

[11] A. Shayan, CSIRO, Melbourne, Australia. Personal communication.

[12] S. Greg MacNeill, Jacques Whitford & Associates Limited, Halifax, Nova Scotia, Personal communication.

[13] Grattan-Bellew, P.E., Is High Undulatory Extinction in Quartz Indicative of Alkali-Expansivity of Granitic Aggregates, Proc. 7th International Conference on Alkali-Aggregate Reaction, Editor, P.E. Grattan-Bellew, Noyes Publications, Park Ridge, New Jersey, USA, 434-439, 1987.

[14] C.A. Rogers, Ministry of Transportation, Engineering Materials Office, Downsview, Ontario. Personal communication.

[15] Berard, J., & Lapierre, N., Reactivites aux alcalis du Gres de Potsdam dans les bétons. Canadian J. of Civil Engineering, 4, 3, 332-344, 1977.

[16] John A. Morrison, Alberta Environment, Edmonton, Alberta. Personal communication.

[17] Bérube, M.A. et Fournier, B., Le barrage Sartigan dans la Beauce (Quebec), Canada: un cas-type de détérioration du béton par des réactions alcalis-granulats, Revue canadienne de génie civil, 14, 3, 372-407, 1987.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

EXAMPLES OF ALKALI-AGGREGATE REACTIVITY IN CONCRETE STRUCTURES IN WESTERN CANADA

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SYNOPSIS:

The recent Canadian Standards Association Supplement No. 2-1986 to CAN3-A23.1-M77 states that "Potentially reactive aggregates occur in most regions of Canada. However, deleterious expansion of concrete containing such aggregates has generally not been observed in Western Canada, where the normal type 10 cement has an alkali content of about 0.7% Na_2O equivalent."

In evaluating potential new sources of aggregate for suitability for use in concrete, using CSA recommended alkali-aggregate reactivity test procedures, certain Southern Alberta aggregates indicated a potential for deleterious expansion. As a result field examinations were conducted on four aged concrete structures in the region, made with similar aggregates. These structures were exposed to external weathering and ranged in age from 37 to 67 years at the time of investigation. A deteriorated, 45 year old concrete reservoir in British Columbia and deteriorated bridge piers in Medicine Hat were also investigated. All of these structures displayed varying degrees of deterioration, ranging from minor localized pattern cracking, to extensive deep-seated pattern cracking, leaching, exudations, scaling and spalling. Petrographic examination of cores extracted from these structures revealed the presence of silica gel and reaction rims around aggregate particles. Most of the concrete examined was non-air entrained and deterioration was attributed to a combination of frost attack and alkali aggregate reactivity. These studies indicate that Western Canada is not immune from AAR and proposed new sources of aggregate should be carefully evaluated, using the Supplement No. 2-1986 to CAN 3-A23.1-M77 procedures, prior to use in concrete.

1.0 INTRODUCTION

Prior to 1940, it was generally assumed that aggregates were an innocuous and inert constituent of Portland cement concrete. It is, however, now recognized that all natural rocks in concrete react with the alkaline pore solution in concrete to a greater or lesser degree. These alkaline components are usually derived from the Portland cement component of the concrete, although other concrete constituents, or alkali migration from external sources can also contribute to the total alkalies in the concrete. While in most cases this alkali aggregate reactivity (AAR) is not harmful, in some instances it can result in deleterious expansion and cracking, with subsequent concrete deterioration.

In several regions in Canada, notably parts of Ontario, Quebec and the Maritime Provinces, testing and evaluation of aged concrete structures in the field over the past 30 years has shown that AAR can give rise to pronounced concrete deterioration, particularly in hydraulic structures or infrastructure subjected to saturation or alternate wetting and drying; e.g. dams, locks, canals, wharfs, piers, bridge decks, highway median barriers and curb and gutter.

As recently as 1986, the Canadian Standards Association (Supplement No. 2 -1986 to CSA Standard CAN3-A23.1-M77) reported that: "Potentially reactive aggregates occur in most regions of Canada. However, deleterious expansion of concrete containing such aggregates has generally not been observed in Western Canada, where the normal type 10 cement has an alkali content of about 0.70% Na_2O equivalent." As a broad generalization, this statement is correct. However, recent field and laboratory studies by Hardy BBT Limited [1] complemented by investigations conducted at the University of Calgary and National Research Council, Institute for Research in Construction, Ottawa, have identified several cases where AAR is considered to have contributed to deterioration observed in hydraulic and highway structures in Alberta and British Columbia.

Review of recent monthly, or yearly average cement test records for Western Canadian cements, indicates that while alkali contents average about 0.64% Na_2O equivalent, they typically range from 0.47 to 0.85% Na_2O equivalent. The lower values are typically found in British Columbia and Southern Alberta cements, while the higher values occur in cements produced elsewhere in the Prairies. Also, recent laboratory studies conducted by Hardy BBT Limited on certain Alberta aggregates, using the AAR test procedures recommended in the Supplement No. 2 to CSA Standard CAN3-A23.1-M77 and the "accelerated South African mortar bar test" have indicated a potential for excessive AAR expansion in aggregates from some sources.

In summary, in view of the fact that:

- cements with alkali contents in excess of 0.70% Na_2O equivalent are being produced in Western Canada;
- testing of aggregates to CSA recommended procedures (and the accelerated South African mortar bar test) has provided evidence of aggregates with a potential for excessive AAR expansion;

- examination of aged concrete structures in the field (including petrographic examination of extracted cores) reveals evidence of AAR;

it is suggested that owners, designers, suppliers and builders of concrete structures in Western Canada cannot afford to adopt a complacent attitude towards AAR. New sources of aggregate, not previously used in Portland cement concrete, and existing but untested aggregate sources should undergo careful evaluation for potentially deleterious AAR prior to use; where excessive reactivity is indicated, alternative sources of aggregate supply should be investigated or appropriate mitigative measures adopted.

This paper briefly reviews six case histories where evidence of AAR was found in aged concrete structures. The six structures are:

- Water Reservoir, Canadian Forces Base, Chilliwack, British Columbia;
- Oldman River Diversion Weir, Fort McLeod, Southern Alberta;
- Oldman River Flume, Fort McLeod, Southern Alberta;
- Lundbreck Falls Bridge, Highway No. 3, Pincher Creek, Southern Alberta;
- St. Mary's Reservoir Spillway, Fort McLeod, Southern Alberta;
- Finlay Bridge, Medicine Hat, Southern Alberta.

2.0 EXAMPLES OF AAR IN CONCRETE STRUCTURES IN WESTERN CANADA

2.1 Water Reservoir, Canadian Forces Base, Chilliwack, B.C.

In 1986, Hardy BBT Limited conducted an investigation of a deteriorating 200,000 gallon water reservoir at the Canadian Forces Base in Chilliwack, B.C. [1]. The reservoir was constructed in the early 1940's and was about 45 years old at the time of evaluation. The reservoir was constructed with reinforced concrete walls, strengthened by exterior pilasters on the east and west walls and buttresses on the north and south walls. Figure 1 provides a general view of the south wall of the reservoir. Figure 2 is a close-up view of a core hole in a buttress at the southwest corner of the reservoir.

Numerous horizontal and vertical cracks and extensive pattern cracking was observed in the walls, pilasters, and buttresses. Figure 2 shows the pronounced pattern cracking in a buttress, with leachate deposits. Concrete at this location was easily removed with a geologist's hammer. Extracted core samples were submitted to the Institute for Research in Construction in Ottawa for detailed petrographic examination. They reported that the cores showed evidence of both frost damage and alkali aggregate reaction. The parameters of the air void system were not determined, but it is likely that the concrete was not air entrained, since air entraining admixtures were not yet in general use in Canada at the time the reservoir was constructed.

Petrographic examination identified the main coarse aggregate components as fine-grained volcanic rocks with a matrix containing micro-crystalline quartz and feldspar microliths, with occasional chert particles. Some particles of coarse-grained granitic rocks which contained strained or micro-crystalline quartz, were also identified.

These rock types are considered potentially reactive. The fine aggregate was comprised predominantly of quartz grains, feldspar, fine-grained altered volcanic rocks and chert. It should be noted that even small amounts of chert (3 to 5%) may cause deleterious expansion in concrete.

Examination of polished sections of cores under a binocular microscope revealed deposits of gel:

- in air voids;
- in cracks within aggregate particles, some of which extended through into the paste matrix;
- in cracks around the perimeter of debonded aggregate particles.

These features are all characteristic of AAR. The Institute for Research in Construction report [1] noted that while alkali-aggregate reaction is not normally encountered in Western Canada, due to the use of low alkali cement, in the case of this reservoir the reaction was probably triggered by the use of an alkali sensitive aggregate in an almost continuously moist environment. The report concluded that the cracking in the concrete reservoir is probably due to a combination of alkali aggregate reaction and frost action.

2.2 Oldman River Diversion Weir, Fort McLeod, Southern Alberta

In 1974 and 1976, Hardy Associates conducted an investigation of the condition of the Lethbridge Northern Irrigation District Oldman River diversion weir and gate support structures on behalf of Alberta Environment [1]. The concrete in this structure was originally placed in 1921. Figure 3 shows a general view of the structure as it existed in 1974. Varying degrees of cracking and deterioration, described as varying from relatively minor hairline cracking (pattern cracking), to wide structural (thermal and shrinkage cracks) were reported to exist in the gate support structure.

A number of the piers were reported to display varying degrees of cracking and surface and edge spalling particularly in locations vulnerable to saturation. Pier concrete at the water level, which was subjected to both saturation and erosion abrasion, was reported to be in very poor condition in a number of the piers. Extensive coring and non-destructive testing was used to quantify the condition of the structure. A concrete core evaluated according to the ASTM C457 test procedure for parameters of the air void system verified (as expected) that the concrete was non-air entrained. Test results are given in Table 1. The report concluded that much of the observed distress was attributable to freeze/thaw deterioration of components of the structure vulnerable to critical saturation during freezing and thawing. In the 1980's, an extensive remedial program was undertaken to rehabilitate the deteriorated parts of the structure.

In 1988, the repaired structure was again examined [1]. The only part of the original concrete structure still readily accessible to examination was a part of the left abutment wall (looking downstream) which had not been repaired. The concrete was observed to be in a fairly good condition although it did display come deep-seated pattern cracking. Cracks both vertical and sub-parallel to the surface were evident. The

fairly good condition of this wall, relative to the original piers in the river, is likely due to the relatively dry exposure condition of the concrete at this location.

Gillott [1] conducted detailed petrographic examination of cores from this wall. Rims were found surrounding many aggregate particles, and white deposits in fractures and voids within aggregate particles and the paste matrix were also found. Microscopic examination of crushed material in polarized light and in liquids of known refractive index showed that silica gel and carbonate materials were both present in these white deposits. Figure 4 shows a crack in a core from this structure. The crack is in a chert aggregate containing carbonates and is filled with silica gel.

Thin section examination of the cores by Gillott [1] showed that the coarse aggregate was comprised of coarse-grained greywacke, chert and smaller amounts of limestone, carbonate-cemented sandstone and igneous rock. The greywacke contained feldspar, chert, quartz, carbonate and recrystallized shale in a matrix of chlorite, sericite and fine silica. The sand fraction consisted of grains of quartz, rock fragments and chert. It was concluded that the formation of silica gel was associated with chert, greywacke and carbonate-cemented cherty sandstone. Gillott concluded that "there is no doubt that alkali aggregate reaction has occurred and must be considered as a factor in the deterioration of the concrete."

2.3 Oldman River Flume, Fort McLeod, Southern Alberta

In 1974 and 1976, Hardy Associates conducted an investigation of the condition of the pier structures supporting the Lethbridge Northern Irrigation District flume crossing the Oldman River. The flume was built in the 1920's. Figure 5 illustrates the condition of pier No. 2 in 1976. The condition of the concrete between the various piers varied considerably, depending on the degree of saturation to which the piers were subjected (either from the river or the leaking flume). Non-destructive testing techniques (impact hammer, Windsor probe, ultrasonic pulse velocity testing), complemented by extensive coring, revealed that:

- Much of the outer layer of surface concrete (to a depth of up to about 15 mm) was soft.
- The concrete was severely cracked in planes both parallel and perpendicular to the concrete surfaces.
- No sound cores could be extracted, even at the centre of the concrete sections.

Petrographic examination of extracted cores revealed extensive cracking with weak outer layers and rims in many otherwise strong coarse aggregate particles. The coarse aggregate was comprised predominantly of quartzite, calcareous sandstone and siltstone, with occasional crystalline carbonates. Some weak ironstone, sandstone and mudstone particles were also noted. Two concrete cores were extracted from the piers for determination of the parameters of the air voids system. Test results are given in Table 1. Clearly, the concrete is non air-entrained. The report concluded that most of the concrete was in a poor condition and attributed

the observed distress primarily to freeze-thaw action and thermal stresses.

The pier structures were demolished in the 1980's and the concrete rubble buried. It was thus not available for examination in 1988. The nature of the observed deterioration, however, indicates that while freeze/thaw attack of this non-air entrained concrete was likely the main cause of distress, AAR may also have contributed to the deterioration of the pier concrete over the 60 years it was in service.

2.4 Lundbreck Falls Bridge, Highway No. 3, Southern Alberta

In 1988, Hardy BBT Limited and Gillott [1], conducted a visual examination of the condition of the abutments at the Lundbreck Falls Bridge on Highway No. 3 in Southern Alberta. Cores were subsequently extracted from the east abutment for petrographic examination. Various bridge structures have been constructed at this site over the years, but the existing bridge structure was constructed in 1953. Various degrees of distress were noted in different components of the structure. Remnants of some of the earliest abutment structures had disintegrated into rubble. The abutment structures constructed in 1953 were providing their intended function, but did display deep-seated pattern cracking.

A core extracted from the west abutment was examined for parameters of the air voids system. Test results are given in the appended Table 1. The concrete in this structure appears to be non air-entrained, and it is likely that much of the deterioration observed can be attributed to frost attack. This is particularly true of curb and gutter and other components of the bridge subject to exposure to deicing salts.

Petrographic examination by Gillott of concrete cores extracted from this structure revealed the following:

- Extensive cracking, both perpendicular and parallel to the exposed surface;
- Cracks which followed the cement-aggregate interface, causing bond failure, as well as passing through aggregate particles and the cement paste;
- Reaction rims with associated white deposits on coarse aggregate particles;
- Deposits of silica gel in cracks in aggregate particles (see Figure 6).

In thin-section, the coarse aggregate was found to be gravel composed of limestone, chert and greywacke. The limestone lithology varied from coarse-grained and fossiliferous to fine-grained and argillaceous; some of the limestone was dolomitic. The chert was microcrystalline and contained patches of chalcedony and carbonate. The greywacke was comprised of strained quartz, chert and carbonates in a matrix of sericitic and clayey material. Quartzite and carbonate cemented sandstone were also present. The fine aggregate was comprised of grains of quartz, carbonates, chert and greywacke type rock fragments.

Gillott noted that concrete from the Lundbreck Falls abutment was the most severely deteriorated of any of the cores from the four Southern Alberta structures he examined. AAR was considered to have contributed to the deterioration of this concrete. It should, however, be noted that this bridge has been in service for 35 years and is still in use.

2.5 St. Mary's Reservoir Spillway, Fort McLeod, Southern Alberta

In 1988, Hardy BBT Limited and Gillott [1] conducted a visual examination of the condition of concrete in the St. Mary's Reservoir Spillway. Additional coring and non-destructive testing was conducted in 1989. This structure was constructed during 1951 and 1952. The concrete placed in 1951 was not air entrained. Air entrainment was used in the concrete placed in 1952, but construction reports indicate that some difficulty was experienced in achieving consistent air contents with the materials available at that time.

Investigations on this structure were concentrated on the piers which support the spillway gates and road structure, and the spillway retaining walls. Tests conducted included in situ, non-destructive testing (impact hammer, Windsor probe, ultrasonic pulse velocity) half-cell potential measurements and chloride ion profiling. Cores were extracted and tested for compressive strength, density, ultrasonic pulse velocity and parameters of the air voids system. The concrete in the core tested for air voids was not air entrained, as shown in Table 1.

Some minor map cracking was noted at the base of some of the piers, which were subjected to saturation from water seeping under the gate stop logs. Map cracking was noted at some locations in the spillway retaining walls, as illustrated in Figure 7. Cracking and concrete deterioration and spalling was noted at the base of joints in the spillway walls, in locations vulnerable to leakage and saturation.

Petrographic examination of cores from this structure by Gillott and the Institute for Research in Construction [1] provided evidence of cracking in the aggregate particles and in the mortar. The development of reaction rims around reactive aggregate particles and the presence of gel in air voids and cracks was recorded. Figure 8 illustrates a rimmed aggregate particle with a white deposit in an adjacent void. A wide range of rock types were observed in the cores. The dominant rock types were chert, jasper and sandstone, with lesser amounts of granite, gneiss volcanics, limestone and quartzite.

It was concluded by the Institute for Research in Construction [1] that the presence of cracks in the mortar phase, the presence of gel partially filling cracks, the occurrence of reaction rims and debonding around some aggregate particles, all pointed to alkali-silica reaction as the probable cause of deterioration of the concrete. The lack of air entrainment, and frost attack likely also contributed to the observed deterioration. It should, however, be noted that the majority of the structure (which was not subjected to saturation) was still in reasonably

good condition, and has provided its intended function over the 37 years it has been in service.

2.6 Finlay Bridge, Medicine Hat

In 1989, Hardy BBT Limited conducted an evaluation of the condition of the piers of the Finlay Bridge in Medicine Hat, Southern Alberta. A general view of one of the piers is shown in Figure 9. Extensive deep-seated pattern cracking and some abrasion erosion deterioration is evident near the water level. Of more concern is the deep-seated pattern cracking in the pier cap, as shown in Figure 10. Evaluation of the piers included: visual examination and photographic documentation of the condition of the pier concrete; non-destructive testing by impact hammer, Windsor probe and ultrasonic pulse velocity methods; extraction of cores for determination of compressive strength, density, air void analysis and petrographic examination.

Strength testing indicated that compressive strength of uncracked in situ concrete was around 35 MPa. The concrete was non-air entrained, as shown in Table 1. Petrographic examination revealed extensive reaction rims around coarse aggregate particles, fractured aggregate particles and paste/aggregate debonding. It was concluded that AAR, acting concomitantly with freezing and thawing had caused the pronounced concrete cracking and deterioration observed. This structure is now slated for extensive remedial works.

3.0 CONCLUSIONS

In 1986, the Canadian Standards Association reported that: "Potentially reactive aggregates occur in most regions of Canada. However, deleterious expansion of concrete containing such aggregates has generally not been observed in Western Canada." It is true that prior to 1989, there were hardly any reported incidents of AAR in concrete structures in Western Canada in the literature. Also, Western Canada has not experienced the widespread AAR deterioration which is so evident and has been so extensively documented for other parts of Canada.

It is, however, suggested that the lack of reported incidents of AAR in the literature for Western Canada is not because potentially alkali-reactive aggregates do not exist, but rather because most of the cements produced in Western Canada have typically had low alkali contents relative to most Eastern and Central Canadian cements. Caution is, however, warranted, since not all Western Canadian cements have low alkali contents and with the advent of "Free Trade" and increased appearance of "off-shore" cements in Western Canada, designers should be alert to possible increasing levels of alkalies in cements proposed for use in Western Canada.

Recent investigations of six aged hydraulic and bridge structures in British Columbia and Alberta have provided evidence of concrete deterioration in which AAR is considered to have been a contributing factor. The structures in question varied in age from 35 to 67 years at the time of investigation. One of the structures (LNID Flume, Oldman

River) has been demolished and replaced. Other structures are under consideration for decommissioning (Chilliwack Reservoir), have undergone substantial repair (LNID headworks structure) or will shortly require remedial work (Lundbreck Falls Bridge, St. Mary's Reservoir, Finlay bridge). It should, however, be noted that these six structures have provided a useful service life. Distress has tended to be concentrated in those parts of the structure subjected to saturation. Also, all six structures (with the exception of part of St. Mary's reservoir) were designed in accordance with prevailing engineering practice, which did not include the use of air entraining admixtures. Freeze/thaw attack has undoubtedly also played a major role in the observed deterioration in all of these structures.

These six case histories provide a warning to owners, designers, suppliers and builders that the potential for problems attributable to AAR exists in concrete structures in Western Canada. This is particularly true for hydraulic structures and other structures subjected to saturation in aggressive external environments, and where the design life of the structure is 30 years or more. New sources of aggregate, not previously used in concrete, should be carefully evaluated for potential AAR, using the procedures detailed in the National Standard of Canada, CAN 3-A23.1-M77, Supplement No. 2, 1986. Sufficient lead time should be provided to enable the tests to be completed prior to the use of the aggregate in concrete construction. Where excessive AAR is indicated, alternative sources of aggregate supply should be investigated or suitable mitigative measures implemented at the design stage.

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REFERENCES

[1] Morgan, D.R. and Empey, D. "Alkali Aggregate Reactivity in Concrete Structures in Western Canada", Canadian Association of Dam Safety Officials, Dam Safety Seminar, Edmonton, Alberta, September 27-29, 1989, pp. 91-121.

TABLE 1:
Parameters of the Air Voids System in Five Aged Concrete Structures in Alberta

Structure	Location	Year Constructed	Air Voids (%)	Specific Surface mm-1	Spacing Factor mm	Density kg/m3
Lundbreck Falls Bridge	Pilaster Northwest Abutment Wall	1953	2.17	8.0	0.90	2445
St. Mary's Reservoir Spillway	Pier Supporting Road Structure	1951	0.88	30.6	0.41	2362
LNID Diversion Weir Oldman River	Northwest Bank Abutment Wall	1922	0.99	16.2	0.67	2361
LNID Flume Oldman River	Pier Structure	1921	2.56 1.28	7.9 9.3	0.82 0.79	2403 2467
Finlay Bridge	North Pier	-	1.41	13.2	0.61	-

NOTE: These parameters of the air voids system indicate that the concrete in all five structures at the location tested was non-air entrained.

FIGURE 1: General view of 200,000 gallon reservoir at Armed Forces Base, Chilliwack, B.C.

FIGURE 2: Close-up view of a deteriorated buttress in the reservoir shown in Figure 1.

FIGURE 3: General view of the LNID Oldman River diversion weir and gate support structure in 1974.

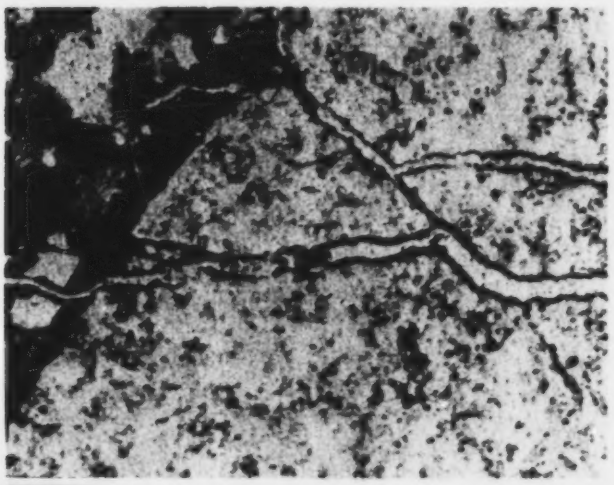

FIGURE 4: Cracks filled with gel in chert containing carbonates; core from abutment wall on northwest bank of LNID headworks structure, Oldman River.

FIGURE 5: Oldman River flume; condition of pier No. 2 in 1976.

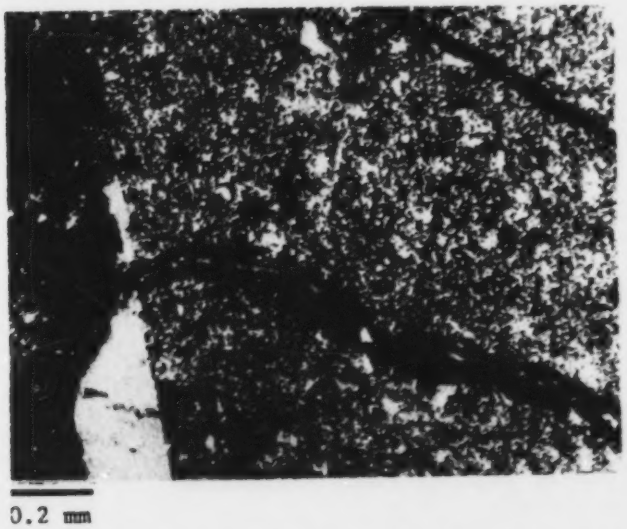

FIGURE 6: Optical micrograph of gel in a crack in cherty aggregate; core from left abutment of Lundbreck Falls bridge near Pincher Creek.

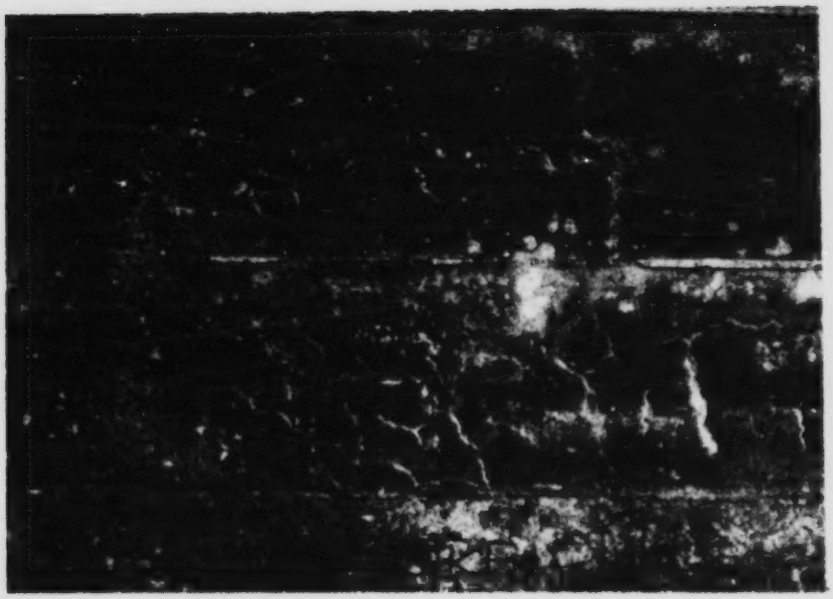

FIGURE 7: St. Mary's Reservoir Spillway; map cracking in spillway retaining wall.

FIGURE 8: Rimmed aggregate particle with white deposit in adjacent void; core from St. Mary's Reservoir Spillway.

FIGURE 9: Finlay Bridge; general view of map cracking and abrasion erosion deterioration in pier structure.

FIGURE 10: Finlay Bridge; close-up view of deep-seated pattern cracking in a bridge pier cap.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

AAR RESEARCH FOR CONCRETE STRUCTURES IN NOVA SCOTIA

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1. Introduction

Nova Scotia has been long identified as possessing aggregates which are potentially alkali-aggregate reactive (AAR). Specifically, greywacke aggregates of the Halifax area are now proving to be most reactive, and the long term damage caused by their expansion will require a considerable effort to effect repairs.

There are over forty concrete ready mix suppliers in the Province and the major suppliers are located in the Metro Halifax area. Local greywacke quarries supply these ready mix plants and this material is also trucked to plants located some three hundred kilometres away.

The Nova Scotia Department of Transportation and Communications (NSDTC) is a major user of ready mix concrete. Since the early sixties, the construction of our arterial highway system has necessitated the building of many structures which are recognized to be alkali-aggregate reactive. Compounding this problem is our marine environment and our current snow removal policy which requires large applications of salt to produce a bare pavement condition within six hours of a major snowfall.

2. Geology

The rock type that has been identified with severe AAR are the quartzites (greywacke). This rock was formed in the Paleozoic Era during the Ordovician Period and known as the Goldenville Formation of the Meguma Group. The individual quartzite beds are interlaminated with narrow bands of slate making up about 5%. The Goldenville quartzites have been folded into a northeasterly striking system of parallel folds that plunge both northeast and southwest. These folds are extensively faulted. It is thought that the quartzite is 4.8 km in thickness. The quartzite or greywacke is light grey to dark greenish grey, breaks with a conchoidal fracture, and commonly grades into narrow bands of micaceous, siliceous slate. Greywackes are dense, uniform textured, chloritic, fine-grained rock that characteristically weathers light gray to white in colour. Some beds contain pyrite which occurs along the bedding planes and imparts a brown colour to the weathered rock.

The greywacke consists of angular to subrounded quartz fragments, with a few feldspar fragments. The feldspar content is less than 10 %, whereas the quartz content may be as high as 85 %.

The formation has been affected by various degrees of metamorphism and have been well compacted and therefore, are quite hard. Distinct schisosity is present and in cases where folding is severe, chlorite schists and mica schists exist.

3. Early AAR Research

Our first indication that some of our aggregates may be of AAR potential was a study conducted during the late 1960's by Duncan, Swenson, Gillott and Foran [1]. This five year study examined over 54 aggregates and used three different cements with varying alkali contents. Three different methods were used to determine expansion and included :

i) modified mortar bar test
ii) concrete prism method
iii) rock cylinder tests

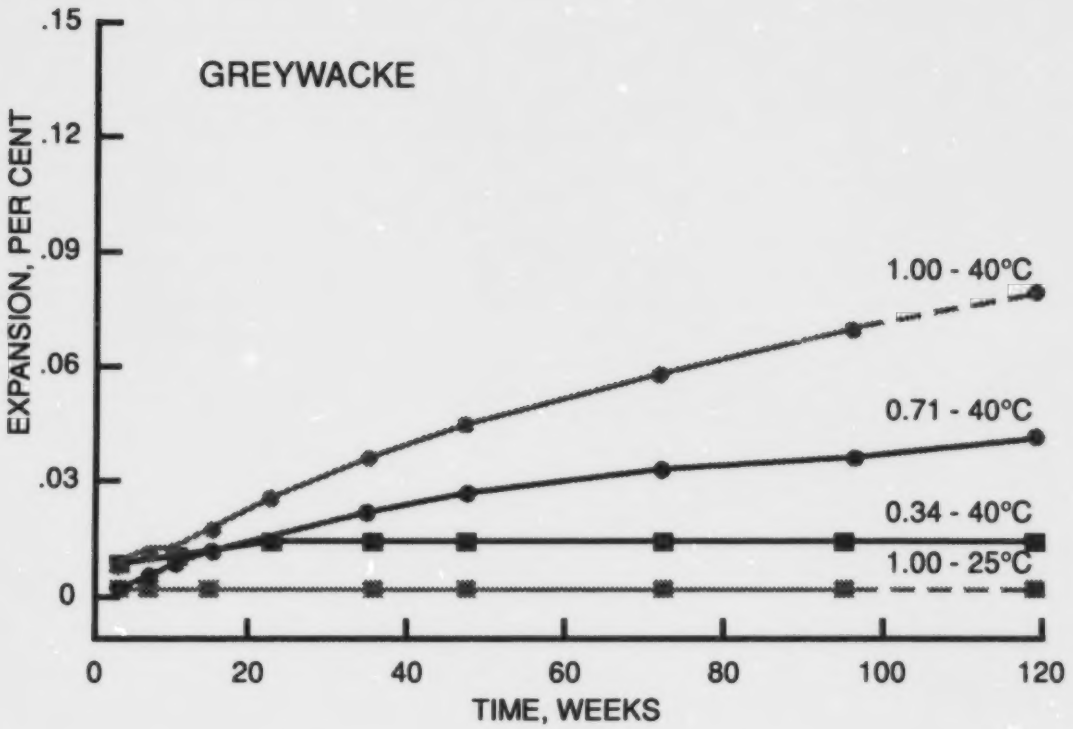

FIGURE 1
LINEAR EXPANSION OF CONCRETE PRISMS

Results of this study clearly indicated that aggregates with AAR potential existed. Of particular interest was the greywacke formation which was identified earlier is a major aggregate source for NSDTC. The study indicated that expansion occurred at a slow rate and was dependent on the available alkalis and moisture. In addition, temperature played a vital role in the ability to reproduce expansions in a reasonable period. As shown in Figure 1, greywacke was identified as a potential AAR material and the significance of the temperature of testing was also important.

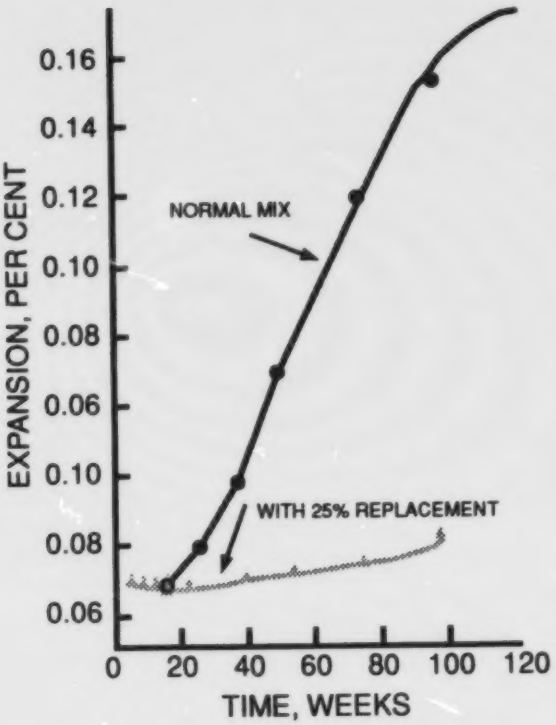

FIGURE 2
EFFECT OF FLY ASH
ON LINEAR EXPANSION

A secondary part of the study included the use of an active pozzolanic material and a fly-ash to reduce AAR activity. Significant reduction in expansion was obtained but these materials were not available locally. Figure 2 illustrates the important effect fly ash has on reducing AAR activity.

4. 1984 AAR Research By NSDTC

As a result of the previous study, NSDTC initiated an internal study [2] to determine if indeed a problem with AAR existed. Seven different coarse aggregates which represented the majority of aggregates used in the Province were utilized as well as one from Ontario. Our normal Type 10 cement was used in the study and its properties can be found in Table 1.

Table 1
Chemical Composition of Cement, %

Composition	Type 10	Type 30
SiO_2	20.22	20.25
Al_2O_3	5.53	5.59
Fe_2O_3	2.12	1.98
CaO	62.99	62.20
MgO	1.36	1.39
SO_3	3.92	5.35
Na_2O	0.12	0.02
K_2O	1.18	1.26
Na_2O (equivalent)	0.91	0.86
C_3S	51.4	43.7
C_2S	19.2	25.1
C_3A	11.1	11.5
C_4AF	6.5	6.0
Loss on Ignition	2.34	1.50

The concrete prisms were made according to Canadian Standards Association Standard CAN3 - A23.2 - M77.14a and the expansion was measured for a period of 646 days. All concrete prisms did not exceed 0.01 % expansion at six months. The conclusions drawn from this report was that our aggregate expanded at a slow rate and did not appear to be expansive. The relatively low alkali content of the cement coupled with the nature of our aggregates indicated that this test procedure did not fully indicate the full AAR potential.

5. 1986 AAR Research

As a result of the 1984 study, it was decided to continue the research using an accelerated test [3] stored at a temperature of 38° which through the addition of sodium hydroxide raised the alkali content to 3%. Six coarse aggregates were used and the fine aggregate was a limestone from New Brunswick which was neutral to AAR. The properties of the coarse aggregate can be found in Table 2 while the properties of the fine aggregate can be found in Table 3. The cement used was a Type 10 cement with an alkali content of 0.6% (sodium oxide).

Table 2
Properties of Coarse Aggregates

Origin	Petrology	Density, kg/m^3	Absorption, %	Rodded Unit Mass, kg/m^3
Brookville	Limestone	2 833	0.26	1 672
Folly	Granite Diorite	2 722	1.00	1 653
Meadows	Granite Quartz Sandstone	2 742	0.80	1 629
Municipal	Greywacke Shale	2 716	0.60	1 553
Plymouth	Greywacke Granite Sandstone	2 787	1.21	1 586
Porcupine	Granite Diabase	2 662	1.20	1 514

Table 3
Properties of Fine Aggregate

Origin	- Brookville, NB	Absorption, %	- 1.54
Petrology	- Limestone	Fineness Moduless	- 2.84
Density, kg/m^3	- 2 814		

As a secondary part of the test, Class "F" fly ash from the Nova Scotia Power Corporation's power plant in Lingan, Nova Scotia was added to determine its effect on any expansion present. The properties of the fly ash can be seen in Table 4. Cement was replaced by 10 and 15 percent of cementitious fly-ash.

Table 4
Properties of Fly Ash

Chemical Content, %	
Silicon Dioxide	42.77
Aluminum Oxide	25.00
Iron Oxide	21.26
Sulfur Trioxide	0.54
Magnesium Oxide	1.01
Ignition Loss, %	
500 °C	1.14
1000 °C	2.65
Available Alkalis, %	0.76
Fineness Pass 350, %	87.55

The batch quantities for the each concrete mix can be seen in Table 5. The concrete was batched with a slump of 75 mm and the air averaged 2.4 %.

Table 5
Mix Properties

Aggregate	Batch Quantities, kg/m³				
	Cement	Fly Ash	Water	Stone	Sand
Brookville	310	-	193	1 028	973
Folly	310	-	193	1 017	905
Meadows	310	-	193	1 002	996
Municipal	310	-	193	956	1 004
Municipal *	279	35	187	956	1 010
Municipal **	264	53	183	956	1 017
Plymouth	310	-	193	975	972
Porcupine	310	-	193	931	1 009

* 10 % fly ash
** 15 % fly ash

As shown in Figure 3, the Municipal aggregate finally showed its AAR colours. This greywacke aggregate exceeded expansion limits while the other aggregates as shown in Figure 4 did not show significant expansion throughout the 280 day testing. The effect of fly ash also was evident as the replacement of 15% cement permitted the test prism to stay within allowable guidelines for expansion.

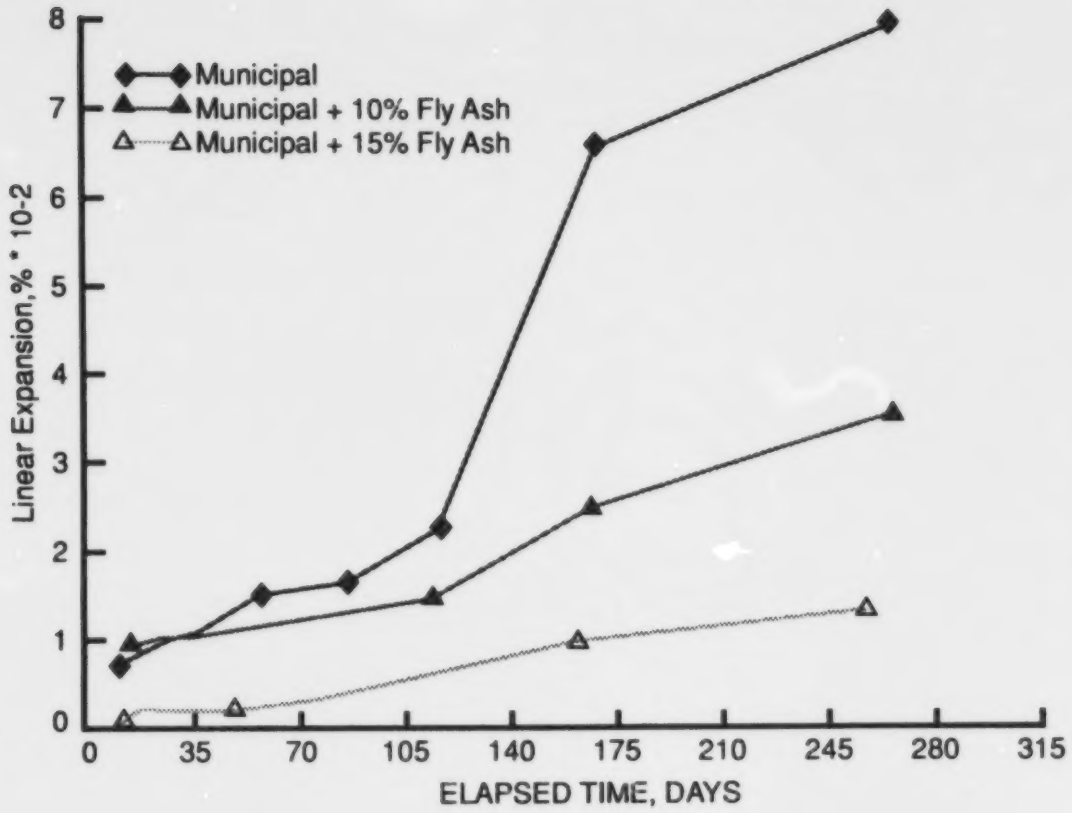

FIGURE 3
REACTION OF MUNICIPAL AGGREGATE

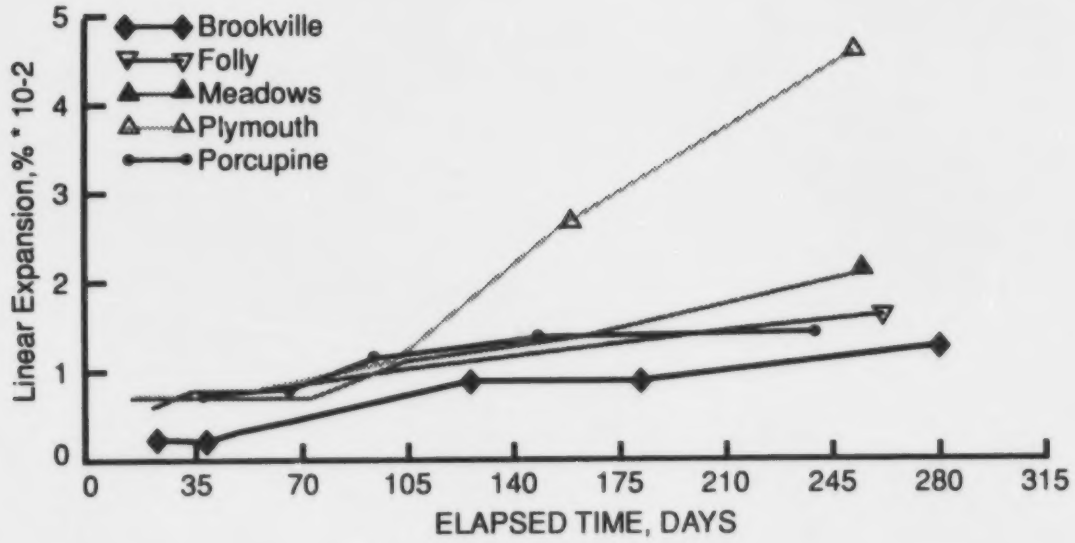

FIGURE 4
OTHER AGGREGATE REACTION

6. NSPC Research

The Nova Scotia Power Corporation obtains most of its electricity through the burning of coal. The by-product of this process is a high quality fly ash which they are currently marketing through a private firm. W.S. Langley & Associates, Halifax,N.S. were hired to examine the use of fly ash in concrete with a specific interest in the reduction of the effects of AAR.

Table 6
Properties of Fly Ash, % - ASTM C618

	ASTM C618	Lingan
Silicon Dioxide		48.75
Aluminum Oxide		23.98
Iron Oxide		17.93
Total	70.0 min	90.66
Magnesium Oxide	5.0 max	1.44
Sulfur Trioxide	5.0 max	0.43
Available Alkalies as NaO	1.5 max	0.235
Moisture Content	3.0 max	0.06
Loss on Ignition	12.0 max *	2.56

* to be lowered to 6 %

Table 6 shows the test results of the fly ash when tested according to ASTM C618. Its high quality can be verified in the low percentage of loss of ignition. Figure 5 clearly indicates the effectiveness of fly ash in reducing the expansion of the concrete. A fly ash content of 15 - 20 percent would seem to promote the best characteristics.

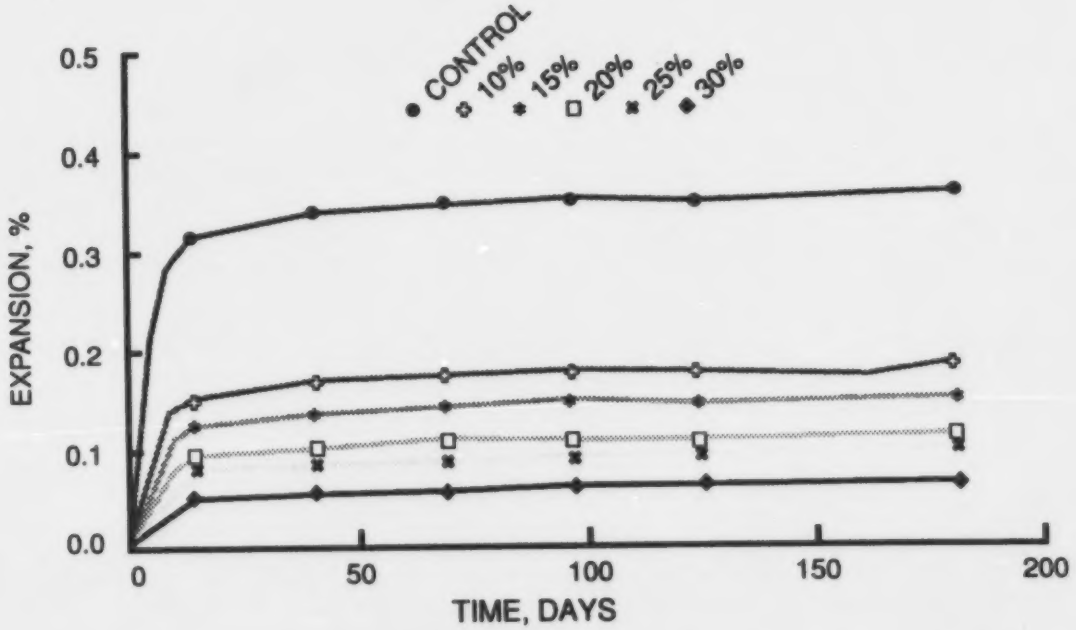

FIGURE 5
ASTM C441, LINGAN FLY ASH

7. In Search of Field AAR

As a result of AAR research conducted by W.S. Langley & Associates and CanMet, a field study was initiated in 1989 by NSDTC to evaluate the condition of structures built with the greywacke aggregate. A total of 47 structures were visually evaluated and classified according to the significance of their AAR activity and age of the structure.

Table 7
Visual Analysis of AAR Structures

Approximate Age, yrs	Number Evaluated	Condition Rating Severe	Moderate	Slight
20	23	9	13	2
15	11	1	9	1
10	7	0	1	6

Note : Structures < 10 yrs all had slight or no AAR.

Table 7 summarizes the data and it is interesting to note that only one of the structures that is 15 years old is severely affected by AAR and none of the structures that are 10 years old exhibited severe damage.

From this visual study, it is evident that moisture and salt play an important role in AAR activity as many of the structures with severe classification can be found on heavily salted routes.

8. NSDTC Response to the AAR Problem

NSDTC had not been an enthusiastic supporter of fly ash as an additive to concrete as we were suspicious of its long term durability. Past tests that were done internally indicated a severe scaling potential. In the past several years, local municipalities have used fly ash in sidewalks and curbs with minimal scaling damage. Our examination of these sites, has indicated that the scaling which did occur was only at the surface and this deterioration can be controlled with sealers if necessary.

We now feel strongly that fly ash will provide the necessary deterrent to the expansive aggregate problem and to that end we are currently revising our specification to permit the use of a maximum of 15 % replacement of cement with fly ash.

9. Our Mounting Repair Problems

Although the inclusion of fly ash in concrete will hopefully stop damage to new structures, the associated repair problems with existing AAR structures will be a problem of the 90's. Our only attempt at AAR repairs was conducted on the Bayers Road Interchange Structure and consisted of removing the deteriorated concrete and patching with latex modified concrete. Three years later, the concrete patches are "popping off" and more rigorous repairs will have to be initiated.

REFERENCES

[1] Duncan, M.A.G., Swenson, E.G., Gillott, J.E., Foran, M.R., Alkali-Aggregate Reaction in Nova Scotia Concretes, Nova Scotia Project Committee on Concrete, September 1970.

[2] Gervais, F.A., Apostolides S.S., Potential Alkali Reactivity of Concrete Aggregates to Nova Scotia, NSDTC internal report, May 1984.

[3] Gervais, F.A., Apostolides S.S., Accelerated Alkali Aggregate Reaction Test, NSDTC internal report, January 1986.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

IDENTIFICATION AND CHARACTERIZATION OF ALKALI SILICATE REACTIVE ARGILLITES AND GREYWACKES IN ATLANTIC CANADA

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1. SYNOPSIS

The distribution and composition of alkali silica gels in field and laboratory concrete samples was studied with argillites and greywackes representative of aggregates from New Brunswick and the Halifax area of Nova Scotia. The alkali reaction products were classified as high and low alkali silica gels. Aggregate particles containing high alkali silica gels were identified as sufficiently reactive to cause excessive expansion. Relationships between the rock composition, mineral grain size, rock fabric, content of alkali-soluble silica and the alkali reactivity indicated a correlation between the amount of high alkali silica gel and intensity of the alkali-silicate reaction. A method based on such petrographic and chemical criteria may be developed for quick identification of alkali-silicate reactive rock types.

2. INTRODUCTION

Compositionally and texturally varied argillites and greywackes of Early Palaeozoic age are amongst the major sources of gravel and quarried concrete aggregate in the Maritime Provinces of Canada. Alkali aggregate reaction (AAR) in concretes containing aggregate composed of argillites and greywackes was first recognized by Duncan [1] in the 1960's in Nova Scotia. The reaction, involving greywackes and argillites, was characterized in 1973 by Gillott and Swenson [2] as "slow expanding" alkali silicate reaction. According to Cox [3], 47 per cent of 122 investigated concrete structures in New Brunswick had developed AAR.

Extensive field survey of structures and laboratory expansion tests of representative aggregates from New Brunswick were carried out during the period 1986-89 as part of the Mineral Development Agreement (MDA) co-sponsored by the Province and the Canada Centre for Mineral and Energy Technology (CANMET). The results obtained from this project and the results of previous work by Gillott and Duncan in Nova Scotia confirm that the argillites and greywackes are responsible for the AAR, however, they also indicate that only some of these rock types are actually reactive.

In this paper, a method for positive identification of reactive and non-reactive particles of argillites and greywackes in field structures and laboratory concrete prisms is presented. The method was developed as a result of both the MDA work and in-house projects at RPC. The results of the preliminary petrographic and chemical investigation of reactive and non-reactive aggregate particles are also presented. The paper lists directions for more detailed work to develop a procedure for quick identification of alkali silicate reactive rocks based on petrographic and chemical criteria.

3. IDENTIFICATION OF REACTIVE AGGREGATE PARTICLES IN FIELD AND LABORATORY CONCRETES

Optical microscope, scanning electron microscope (SEM) and electron microprobe (EPMA) techniques were employed to examine polished thin sections and fractured sections prepared from concretes affected by AAR. Their examination revealed two types of gels; transparent high alkali and milky white low alkali silica gels. As shown by wavelength dispersive spectrophotometry (WDS) analysis*, the CaO/total $NaO+K_2O$ ratio for the high alkali gels has a relatively narrow range of values between 0.7 and 1.0, whereas the CaO/total $NaO+K_2O$ ratio for the low alkali gels ranges from 1.0 to 30.0 as shown in Table 1.

TABLE 1

CHEMICAL COMPOSITION OF ALKALI SILICA GELS

	HIGH ALKALI			/ LOW ALKALI		
Wt.%	1	2	3	4	5	6
SiO_2	61.69	62.56	58.36	64.44	56.8	45.02
Al_2O_3	2.41	0.75	1.34	0.12	0.05	0.27
MgO	0.34	0.09	0.18	0.38	0.00	0.01
FeO	0.66	0.22	0.47	0.09	0.00	0.00
SO_2	0.01	0.03	0.01	0.01	0.01	0.01
CaO	12.85	13.71	13.86	19.49	27.42	40.87
K_2O	10.21	10.42	9.82	6.33	4.04	1.39
Na_2O	5.04	3.61	3.22	4.41	4.04	1.19
Total	93.21	91.39	87.26	95.27	92.40	88.76
CaO/NaO $+K_2O$	0.8	0.9	1.0	1.8	3.3	30.5

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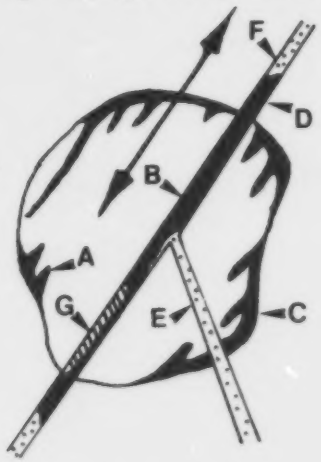
* In order to minimize the sodium and potassium burn-out effect and to obtain good counting statistics on these elements, a 50 nA beam was rastered over a large area of the sample during the analysis. The complete analytical conditions are listed below.

accelerating voltage:	15 kV
probe current:	50 nA
spot size:	scan over 9 mic. X 11.5 mic. area
coating:	carbon
element standards:	Na: albite, 30
and acq. time in	K: sanidine, 30
secs.	Si, Al, Mg, Ca, Fe: almandine, 20

The high alkali gels are found chiefly inside aggregate particles within the "reaction rims". In the rims, gels occur between the grains of rock-forming minerals. Less frequently, the high alkali gels occur on the contacts between aggregate particles and cement paste, in air voids adjacent to aggregate particles and in fractures in the cement paste.

The low alkali gels were observed in fractures dissecting aggregate and cement paste, in air voids, along aggregate-cement paste interfaces and in the form of exudations on the exterior surface of the field structures and laboratory prisms.

Figure 1 shows the distribution of high and low alkali gels inside and around a reactive aggregate particle. The low alkali gels appear to have been formed by alteration of high alkali gels. It is believed that the alteration process involves accommodation of calcium from concrete pore solutions into the structure of high alkali gels exposed to these fluids in open spaces such as fractures and air voids. The process increases the Ca/Na+K ratio of high alkali gels and results in their transparency change from water clear to milky white.



A: high alkali gel, reaction rim
B: high alkali gel, fracture in aggregate particle
C: high alkali gel, surface of aggregate particle
D: high alkali gel, fracture in cement paste
E: low alkali gel, fracture in aggregate particle
F: low alkali gel, fracture in cement paste
G: calcium deposits
* arrow marks foliation of the particle

Fig.1 Distribution of alkali silica gels in and around reactive aggregate particle.

Crystalline precipitates of calcium oxide (hydroxide ?) on the surface of alkali silica gels in the fractures (Fig. 1) and, frequently, the abundant white exudations of calcium

carbonate on the exterior surface of concrete structures both indicate the migration of calcium-rich pore solutions in concrete affected by AAR (surface lime staining can also be from other causes). High alkali gels in fractures and air voids represent, most probably, early formed and unaltered gels that migrated, supposedly as solutions, away from reaction rims into the available open spaces. The studies on synthetic alkali silica gels by Struble and Diamond [4] suggest that liquification of gels takes place in response to stress. It seems probable that both sufficient stress for the liquification of alkali silica gels and a network of fractures can be generated in response to the expansion of high alkali gels formed in the reaction rims of reactive aggregate particles. It is the aggregate particles containing high alkali silica gels that are alkali reactive.

A uniform thickness of reaction rim for a given particle indicates that the major mineral components of the particle are involved in the formation of gels. The SEM/EPMA investigation provides some evidence that, in the rims, the matrix-size quartz grains (finer than 0.006 mm) are replaced by alkali silica gels. It is speculated, therefore, that the chemistry of the alkali silicate reaction is the same as that of the alkali silica reaction, provided that sufficiently high pH environment is created inside the rims.

More work is needed to prove that the concentration of alkalies inside reaction rims results in replacement of quartz by gels. However, the very fact that high alkali silica gels precipitate in the matrix of aggregate particles indicates absorption of alkali-rich fluids into these particles.

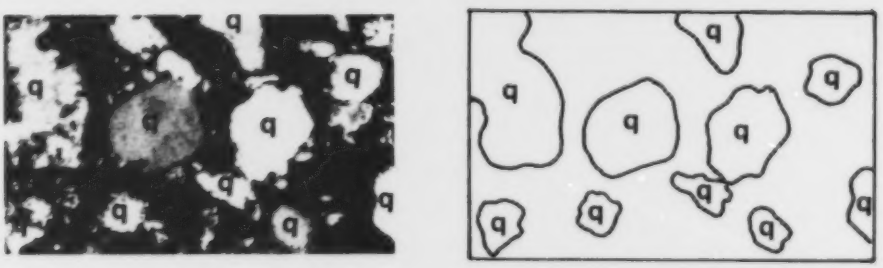
4. MINERAL COMPOSITION, GRAIN SIZE AND METAMORPHIC FABRIC OF REACTIVE AND NON-REACTIVE ARGILLITES AND GREYWACKES

An attempt was made to identify the mineral composition, grain size and fabric (shape and distribution of mineral components due to metamorphism) of aggregate-forming rocks which absorb alkalies and produce high alkali silica gels. Electron microprobe analysis of polished thin sections and x-ray diffraction analysis of pulverized samples show that major constituents of these argillites and greywackes are quartz, chlorite and sericite. These minerals are accompanied by variable (as-a-rule, accessory) quantities of the feldspars, calcite, iron oxides, fragments of other rocks, sphene, biotite, epidote, sulphides, rutile and zircon.

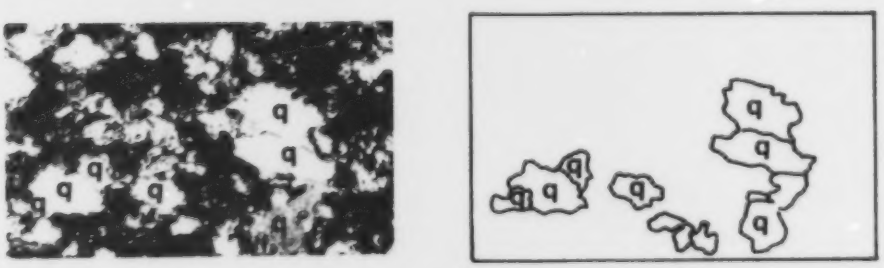
The grain size of chlorite, sericite and quartz in both the argillites and greywackes ranges from below 0.006 mm to 0.5 mm. Rocks containing more than 75% of the material equal to or finer than 0.006 mm are classified as argillites. Greywackes contain more than 25% material coarser than 0.006

mm and finer than 0.5 mm.

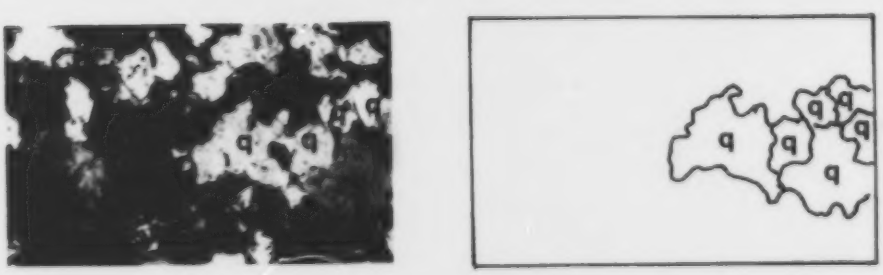
The metamorphic fabric of the greywackes and argillites is classified as slightly, moderately and completely recrystallized based upon the degree of the original shape preserved in grains of quartz from the sediment (Fig. 2). In slightly recrystallized greywackes, most of the quartz grains retain their original shape and only some of them are slightly recrystallized along the edges. All grains of quartz in the moderately recrystallized fabric are recrystallized along the edges, while interlocking fabric characterizes the completely recrystallized sediments.



Slightly recrystallized fabric



Moderately recrystallized fabric



Completely recrystallized fabric

Fig.2 Metamorphic fabric of greywackes in thin sections. Polarized light, magn. X 60. Quartz = (q).

5. AAR PROMOTING FEATURES OF ARGILLITES AND GREYWACKES

Detailed petrographic examination of thin sections prepared from reactive and non-reactive argillite and greywacke particles characterized by slightly and moderately recrystallized fabric was performed. The particles were extracted from 10 samples of field concretes, 11 laboratory concrete prism containing homogeneous argillites and greywackes and 4 laboratory concrete prisms containing heterogeneous argillites and greywackes from quarries or composite aggregates from gravel deposits. The prisms were prepared from cement containing 1.25% alkalies and stored for over two years at temperature of 38 Deg.C and 100% RH in order to induce alkali reactivity of the aggregates. Some observations are reported:

(1). Despite the variation in composition, texture and fabric, both the reactive and non-reactive argillites and greywackes contain quartz, chlorite and sericite as the major constituents. The grain size of the matrix of reactive rocks does not exceed 0.3 mm and only the slightly and moderately recrystallized argillites and greywackes are reactive while their completely recrystallized compositional equivalents are not.

(2). High-grade metamorphic rocks and rocks of granitic or rhyolitic affinity (both with interlocking texture) become reactive if affected by retrograde metamorphism that results in the formation of secondary chlorite, sericite and quartz at the expense of the original minerals and in the modification of the matrix grain size to that characterizing reactive argillites and greywackes (i.e.grain size < 0.3 mm).

(3). A positive correlation exists between the overall abundance of alkali silica gel residing in reaction rims of reactive aggregate particles and expansion rate of the prisms.

(4). Alkali silica gels are produced by temperature dependant chemical reactions as indicated by temperature controlled expansion of the prisms (unpublished data) . Aggregate particles in the prisms stored at room temperatures did not expand.

Based on the results of the examination it was concluded that the rocks composed of fine-grained (< 0.3 mm) chlorite, sericite and quartz contained in slightly and moderately recrystallized matrix may have a unique content of alkali-soluble silica (quartz) that makes them alkali reactive.

6. LABORATORY DETERMINATION OF ALKALI SOLUBLE SILICA

In an attempt to determine the content of alkali soluble

silica in reactive and non-reactive rocks, three samples of greywacke and four samples of argillite of known, from the concrete prism test, reactivity were analyzed using a modified ASTM C 289 procedure [5]. The analysis was performed on 20 grams of pulverized, minus #200 mesh, samples stored for 24 hours at 80 Deg.C in 1.000 mol/l NaOH solution. Table 2 gives the results of the analysis.

The amounts of dissolved SiO_2 shown in Table 2 were determined on a single sample of each rock. Nevertheless, the results differentiate clearly between the reactive (1 - 4) and non-reactive (5 - 7) argillites and greywackes. The reactive rocks contain at least 25% more alkali-soluble silica than their non-reactive counterparts.

TABLE 2

CONTENT OF ALKALI-SOLUBLE SILICA IN ARGILLITES AND GREYWACKES

Sample	Dissolved SiO2 mmoles/l	Expansion % after 2 years (prism)
1. argillite	109.8	0.025
2. greywacke	101.4	0.084
3. greywacke	90.2	0.316
4. argillite	60.5	0.028
5. argillite	45.1	0.008
6. argillite	41.3	0.008
7. greywacke	29.8	0.009

Better correlation between the alkali-soluble silica (quartz) and expansion rate within the group of reactive rocks is expected if a more refined chemical test is applied. The improvements would be aimed at determining the optimum grind of the aggregate needed to liberate the maximum number of alkali-soluble grains of quartz from the rock's matrix and to expose them to the NaOH solution. An extension of this work could be directed to determining if metamorphosed sediments with completely recrystallized (interlocking) fabric contained alkali-soluble silica.

Future studies should also be aimed at determining the minimum, or pessimum, proportion of reactive argillites and greywackes in composite aggregates required to trigger AAR. The current data indicated that the content of argillites and/or greywackes in AAR induced expanded field and laboratory concretes exceeds 65% of the total number of particles in coarse fraction of the aggregate. This figure is substantially higher than that determined for the alkali silica reaction, since only about 4% of chalcedony-like structured silica in

the aggregate appears to be sufficient to expand the confining concrete [6]. It will be worthwhile determining the expansion of a series of concrete prisms containing variable proportions of argillites and greywackes (of known reactivity) mixed with non-reactive rock types, such as granitoids or basalts, known to be unreactive.

7. PROPOSED (PRELIMINARY) RAPID PETROGRAPHIC/CHEMICAL TEST

The proposed petrographic/chemical method for identifying alkali silicate reactive argillites and greywackes, once established, would be as follows:

(1) Determine the mineral composition by x-ray diffraction analysis (are quartz, chlorite or sericite major mineral components ?).

(2) Determine the grain size and metamorphic fabric in thin sections (is the grain size of the groundmass finer than 0.3 mm ?; is the groundmass slightly or moderately recrystallized ?).

(3) Determine the content of alkali-soluble silica using the improved ASTM C 289 method (is it more than 45 mmoles of silica ?).

(4) Determine the percent reactive particles in aggregate, as determined by positive answers to (1)-(3).

8. CONCLUSIONS

(1) Aggregate particles containing high alkali silica gel at grain boundaries of particle-forming minerals are alkali reactive.

(2) Petrographic and chemical data, to-date, on reactive aggregate particles of argillites and greywackes indicates that the reactive rocks are characterized by (1) slightly to moderately recrystallized fabric, (2) mineral constituents grain size that does not exceed 0.3 mm and (3) they contain at least 25% more alkali-soluble silica than the non-reactive rocks.

(3) ASTM C 289 standard chemical test, if modified, may be suitable for determining the amounts of alkali-soluble silica in aggregate.

(4) A combination of petrographic and chemical tests is likely to provide a means for a quick identification method for reactive aggregate.

9. REFERENCES

[1] Duncan, M.A.G., Swenson, E.G., Investigation of Concrete Expansion in Nova Scotia, The Prof. Eng. in Nova Scotia, vol 10, no. 2, May 1969, pp. 16 - 19.

[2] Gillott, J.E., Swenson, E.G., Duncan, M.A.G., Alkali-aggregate Reaction in Nova Scotia, IV, Character of the Reaction, Cement and Concrete Research, 3, 5:521-535, 1973.

[3] Cox, P.E., Alkali Aggregate Reaction in Southern New Brunswick Concrete, Senior Report, Department of Civil Engineering, University of New Brunswick, 1980.

[4] Struble, L., Diamond, S., Unstable Swelling Behaviour of Alkali Silica Gels, Cement and Concrete Research, 11, pp. 611-617, 1981.

[5] ASTM C289-87. Test Method for Potential Reactivity of Aggregates(Chemical Method).

[6] Duncan, M.A.G., Swenson, E.G., Gillott, J.E., Alkali Aggregate Reaction in Nova Scotia, Pt. III, Laboratory Studies of Volume Changes, Cement and Concrete Research 3, 3:233-245, 1973.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

MECHANISM OF EXPANSION IN DUGGAN TEST FOR ALKALI-AGGREGATE REACTION

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SUMMARY

Portland cement concrete suffers durability failure from external attack by weathering agencies and from deleterious internal reactions which include expansive alkali-aggregate reactions (AAR). Recognition of AAR depends mainly on petrographic examination and long-term dimensional change tests. A new test which requires only 6 weeks is being developed at CN-Research. Major objectives of this work are to establish whether expansion measured in the Duggan-test is due to AAR, and if not, to find the mechanisms responsible.

Ten sets of concrete prisms were cast containing combinations of four cements with different alkali contents and, two alkali-expansive and one non-expansive aggregates. Companion samples were subjected to the Duggan-test and the standard concrete prism test (CSA A23.2 - 14A). Comparisons have been made between length change, analytical and petrographic results.

Results show that AAR is not the major cause of expansion measured in the first 21 days of the Duggan-test; expansion depends mainly on properties of the cement. The Duggan-test increased microcracking; microcrack intensity depended on cement type and not on aggregate type. The amount and form of the sulphate minerals in the unhydrated cement may affect the degree of microcracking. The Duggan-test expansion up to 90 days correlates with the relative proportion of ettringite and microcrack frequency in the concrete samples.

1. INTRODUCTION

1.1 Recognition of the problem

Due to concrete durability problems associated with alkali-aggregate reactions (AAR) there is a great need for simple and rapid means of recognition of alkali-expansive aggregates. A new dimensional change test which requires only 6 weeks has been under development at CN-Research. This test involves thermal cycling which probably causes microcracking. Expansion may result from the easy access of alkaline pore solutions to reactive aggregate or from pressure exerted by growth of ettringite or other crystals in the cracks, moisture uptake by siliceous or other gels [1,2] or simply moisture sorption by the concrete.

1.2 Objectives

A major objective of this work is to investigate whether the dimensional changes in the new test are due to AAR or whether there are other reactions which contribute to expansion. The Duggan-test has been performed on cores taken from 10 concrete mixes made with combinations of four cements with different alkali contents and three aggregate types, two alkali-expansive and one non-expansive. The Duggan-test was performed at CN-Research and results are compared with those obtained in the standard dimensional change test (CSA A23.2 - 14A) conducted at The University of Calgary. After-test samples are being subjected to detailed petrographic examination and analysis in an attempt to establish the mechanism responsible for the expansions measured in the Duggan-test.

2. MATERIALS

2.1 Aggregates

Three limestone aggregates were selected. Two are known to be alkali-expansive [3,4] and the third from Exshaw, Alberta, is not alkali-expansive over the time of these experiments. One of the expansive aggregates (Spratt quarry, Ottawa, Ontario) is subject to alkali-silica reaction (ASR) and the other is from the classic location of alkali-carbonate reactive aggregate (Pittsburg quarry, Kingston, Ontario).

Coarse aggregates were crushed and sieved to 3 sizes (14, 10 and 5 mm) and used in the saturated surface dry (SSD) condition. Fine aggregate was prepared from the same rock and used in a dry condition.

2.2 Cements

Three Portland Type 30 high early strength cements were selected on the basis of different alkali contents (Na_2O equiv.). These were named cement 1 (0.46%), cement 2 (0.97%) and cement 3 (1.14%). Cement 1 with alkali content boosted to 1.14% by addition of NaOH to the mixing water was also used.

Table 1: Composition of cements

	Cement:Type 30		
	Cement 1	Cement 2	Cement 3
SiO_2	21.61	20.11	19.97
Al_2O_3	4.39	4.85	4.30
Fe_2O_3	3.89	2.64	2.52
CaO	62.41	62.90	62.26
MgO	3.15	2.10	4.07
Na_2O	0.19	0.30	0.51
K_2O	0.41	1.02	0.96
SO_3	2.92	4.15	4.69
Loss on ignition (L.O.I.)	1.10	2.10	1.10
Free lime	0.51	0.99	1.65
Na_2O equivalent	0.46	0.97	1.14
K_2O: Na_2O	2.16	3.40	1.88
(L.O.I.): free lime	2.16	2.12	0.67

The proportions of free lime and magnesia in the three cements are quite variable. The method of free lime analysis does not differentiate between the unhydrated (CaO) and hydrated ($Ca(OH)_2$) forms, and this is of potential significance in determining the soundness of the cement. However, the loss on ignition value gives an indication of the extent of carbonation and hydration of the free lime and magnesia, and the ratio of loss on ignition to free lime therefore provides an approximate measure of potential soundness. The relatively high free lime content and low loss on ignition of cement 3 suggest that much of the lime is in the unhydrated state, rendering the cement potentially less sound than the others tested.

The Blaine index of all three cements shows that they are of similar fineness the values being: cement 2, 5510 cm^2/g; cement 1, 5200 cm^2/g; and cement 3, 5120 cm^2/g.

2.3 Mix design

A total of ten mixes were cast; nine using all combinations of the three cements and three aggregates, and the tenth with Spratt aggregate and alkali-boosted cement 1. The same aggregate and cement contents were used throughout, and water content was adjusted to give a slump value of around 80 mm. Concrete alkali contents varied from 2.19 to 5.42 kg/m^3 with the different cements.

2.4 Mix strengths

The unconfined compressive strengths of all mixes were determined after 24 hours accelerated curing* and again after fog-room curing for

* Accelerated curing cycle: 2 hours at ambient temp., 2 hours ramp to 85°C, 4 hours at 85°C, cooled to room temp. overnight in oven.

28 days. Mean strengths varied from 37 to 44 MPa at 24 hours, and 56 to 64 MPa at 28 days. Statistical analysis of the results shows that if aggregate type is not taken into account, there is no significant difference between the strength of mixes made with cement 1 and cement 3 or cement 3 and cement 2 at either age. However, at the 95% confidence level, mixes with cement 2 were significantly stronger than those made with cement 1 at both ages.

3. TEST METHODS

3.1 Duggan-test

Each mix was cast as a series of five (3 x 3 x 14 inch) prisms and subjected to accelerated curing. Cores (22 mm diameter) were drilled from one prism and trimmed to 50 ± 5 mm length. Several were sent to the CN-Research laboratory in Montreal for the Duggan-test (Fig.1)[5] and the remainder were stored in distilled water at ambient temperature as control samples. After the Duggan-test two cores of each mix were returned to Calgary for analysis after approximately 21 and 91 days of immersion.

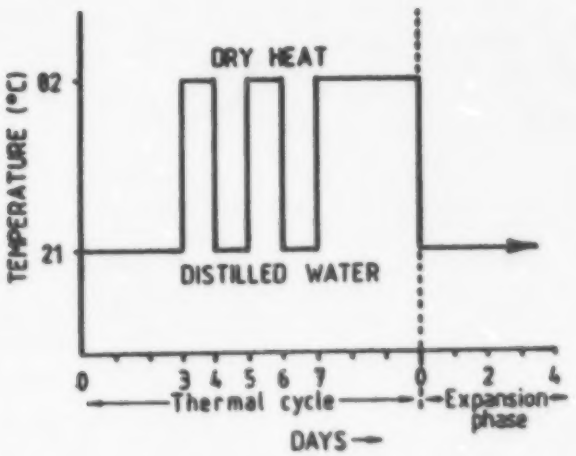

Fig.1. Duggan-test cycle.

3.1.1 Results

All samples expanded during the Duggan-test. The mean expansions of all cores of each mix at each reading were calculated, and results are shown in Fig.2. For each cement type, the pattern of expansion developed with all aggregates was almost identical. Samples made with cement 1 expanded only slightly in the first few days of test, and their dimensions subsequently stabilized, with final levels of strain at around 0.10 to 0.15%. Those made with cement 3 were more expansive,

and developed strains of 0.15 to 0.20% in the first 21 days of immersion. They then continued to expand at a reduced rate throughout the test period, and strains of 0.35 to 0.40% were developed by the end of the experiment. The most expansive samples were those containing cement 2. Although these showed very similar levels of strain to those made with cement 3 after 21 days immersion, they subsequently expanded at a higher rate. At the end of the test mixes made with cement 2 and Exshaw and Spratt aggregate showed strains of about 0.70% and that containing Pittsburg aggregate had expanded by over 0.90%. The pattern of strain shown by samples containing Spratt aggregate and cement 1 with boosted alkali content was similar to that of the other mixes made with unadulterated cement 1, but the final level of expansion was about 0.20%.

3.1.2 Discussion

The nature of the aggregate had little effect on the expansion of samples made with any of the cements during the first 21 days of the test. The magnitude of expansion was not proportional to cement alkali content; this is confirmed by the behaviour of samples containing alkali-boosted cement 1. These results provide very strong evidence that AAR is not the primary cause of expansion in the Duggan-test. However, some petrological evidence of ASR was observed in test samples containing Spratt aggregate (section 4.3.2), and the behaviour of samples made with cement 2 and Pittsburg aggregate suggests that AAR may contribute to expansion in the longer term. It is therefore concluded that the nature of the cement is the primary factor dictating expansion behaviour in the Duggan-test.

3.2. Standard dimensional change test

3.2.1 Results

Companion concrete prisms cast with stainless steel studs at the ends were stored over water in sealed containers at 38°C and expansion was monitored according to CSA A23.2 - 14A. Graphs of length changes up to 90 days are shown in Fig.2.

Expansion was minor in all prisms containing Exshaw aggregate regardless of type of cement and also in prisms containing Spratt aggregate and cement 1. The prism containing Pittsburg aggregate and cement 1 expanded by about 0.07%. The remaining samples, which contain Pittsburg and Spratt aggregates combined with cements 2 and 3, all show larger expansions which range from 0.08 to 0.26%, and are still increasing. In all cases, expansion is greater with cement 3 than with cement 2. Expansion curves given by concretes containing Pittsburg aggregates are concave towards the x-axis whereas concretes containing Spratt aggregates give curves convex towards the x-axis (Fig.2).

3.2.2 Discussion

Results are as predicted from known reactivity of the three aggregates and alkali contents of the cements. With both expansive aggregates the degree of expansion was proportional to cement alkali

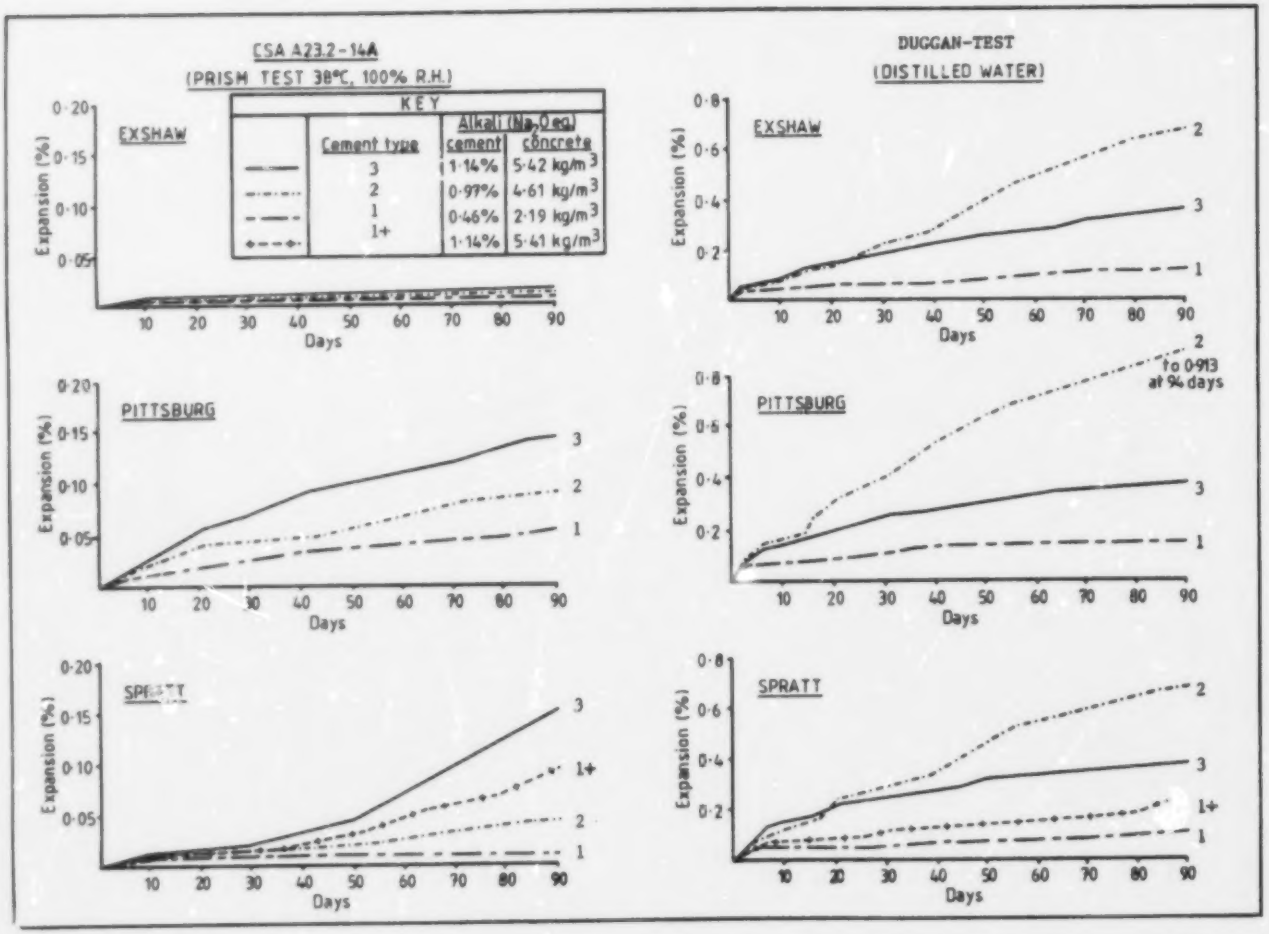

Fig.2. Dimensional change of concrete in Duggan-test compared with CSA prism test.

content. This is confirmed for Spratt aggregate which produced similar expansion in prisms with cement 3, and with alkali-boosted cement 1. Known differences in the style of expansion produced by Pittsburg and Spratt aggregates are confirmed.

4. PETROGRAPHY

4.1. Aggregates

The rock from the Spratt quarry is a coarse to fine-grained calcitic limestone in which about 15% of particles show some dolomitization. The sample used in this study contains 2% or less chalcedony; a few particles of chert are also present. The insoluble residue contains quartz, illite and pyrite. Pittsburg aggregate is a fine- to very-fine dolomitic limestone with an insoluble residue containing detrital quartz, feldspar and clay minerals [3]. The Exshaw limestone is predominantly oolitic with smaller amounts being fossiliferous; it has occasionally been dolomitized. Chert and chalcedony are present in small proportions together with other acid insoluble minerals.

4.2 Cement pastes

The cement pastes of all mixes are normally hydrated, but contain a very high proportion of unhydrated cement clinker attributed to the use of low water/cement ratios. In each case the ferrite phase is distinct, and gives the paste a mottled texture in transmitted light. The SEM reveals all pastes to be dense with relatively low microporosity. All mixes have similar, moderate portlandite contents, but the portlandite crystals in cement 1 are typically smaller and less well defined in thin section than those in the other samples. Portlandite was not observed as pellicles to aggregate particles or in cracks in any of the samples, although the mineral was very occasionally present as large crystals in voids. Significantly more ettringite was observed with the SEM in samples subjected to the Duggan-test than in control samples.

4.3 Deterioration

4.3.1 Cracking

Optical and SEM examinations reveal that in all cases the cement paste is microcracked, but cracking is much more intense in samples after the Duggan-test than before it. Cracks are typically narrow and devoid of infilling, and are usually of very limited extent, passing through the paste between adjacent aggregate particles and around aggregate margins, but rarely passing through the particles themselves. The orientation of the cracks is random, and they are not apparently related to particular aggregate particles.

Microcrack intensity was estimated semiquantitatively by conducting a crack frequency survey on thin sections. The number of microcracks intersected during a series of parallel traverses was

summed, and results were expressed as cracks per metre. Results are highly susceptible to artifacts, operator and optical errors, and are therefore intended to be only an approximate indication of microcrack intensity.

Statistical analysis at the 95% confidence level was performed to assess the significance of differences in the crack frequencies (Fig.3). There is a significant increase in the microcrack frequency in samples which had been subjected to the Duggan heating and cooling cycle and subsequent immersion phase compared to the samples at age 24 hours. Samples were not studied immediately after the thermal cycle so it is not possible to determine whether the increase in microcracking was induced by thermal shock alone, or whether the period of saturation after cycling is important. Leaving the test samples immersed in water beyond the first 21 days did not result in a significant increase in cracking.

Aggregate type had no influence on the degree of microcracking developed at any age. Microcrack frequency in concretes made with cements 1 and 2 was initially low and increased throughout the 21 day period of the Duggan-test, whereas in concretes made with cement 3 it was initially high and did not increase significantly after cycling.

Cracking increased more rapidly in concretes made with cement 2 than in those made with cement 1. At the end of the experiment, the degree of microcracking in concretes made with both these cements had increased to the level which had been present in cement 3 concretes after accelerated curing (age 24 hours).

4.3.2 Alkali-aggregate reaction

No deposits associated with AAR were observed in samples containing Exshaw or Pittsburg aggregates. However, 38°C control samples made with Pittsburg aggregate which have shown expansion in the prism test, were not examined in detail for evidence of cracking. Alkali-silica gel was observed on broken surfaces of control samples containing Spratt aggregate, and traces were also observed in samples subjected to the Duggan-test. The presence of silica gel confirms the occurrence of ASR.

4.3.3 Other processes

Thin section examination indicates that the samples subjected to the Duggan-test had undergone negligible carbonation. The cement paste in all samples showed no evidence of leaching, and secondary portlandite was very rarely observed in cracks or voids. Traces of ettringite were occasionally observed in thin section in microcracks, in samples made with cements 2 and 3 after the Duggan-test, but this mineral was not found in larger cracks or voids. However, large quantities of ettringite were observed with the SEM in the cement paste of these samples. This typically occurred as rosettes of needle-like crystals in pockets and in microporous cement paste, which strongly suggests a secondary origin. The quantity observed in 38°C control samples was much less than that in after-test samples, indicating that

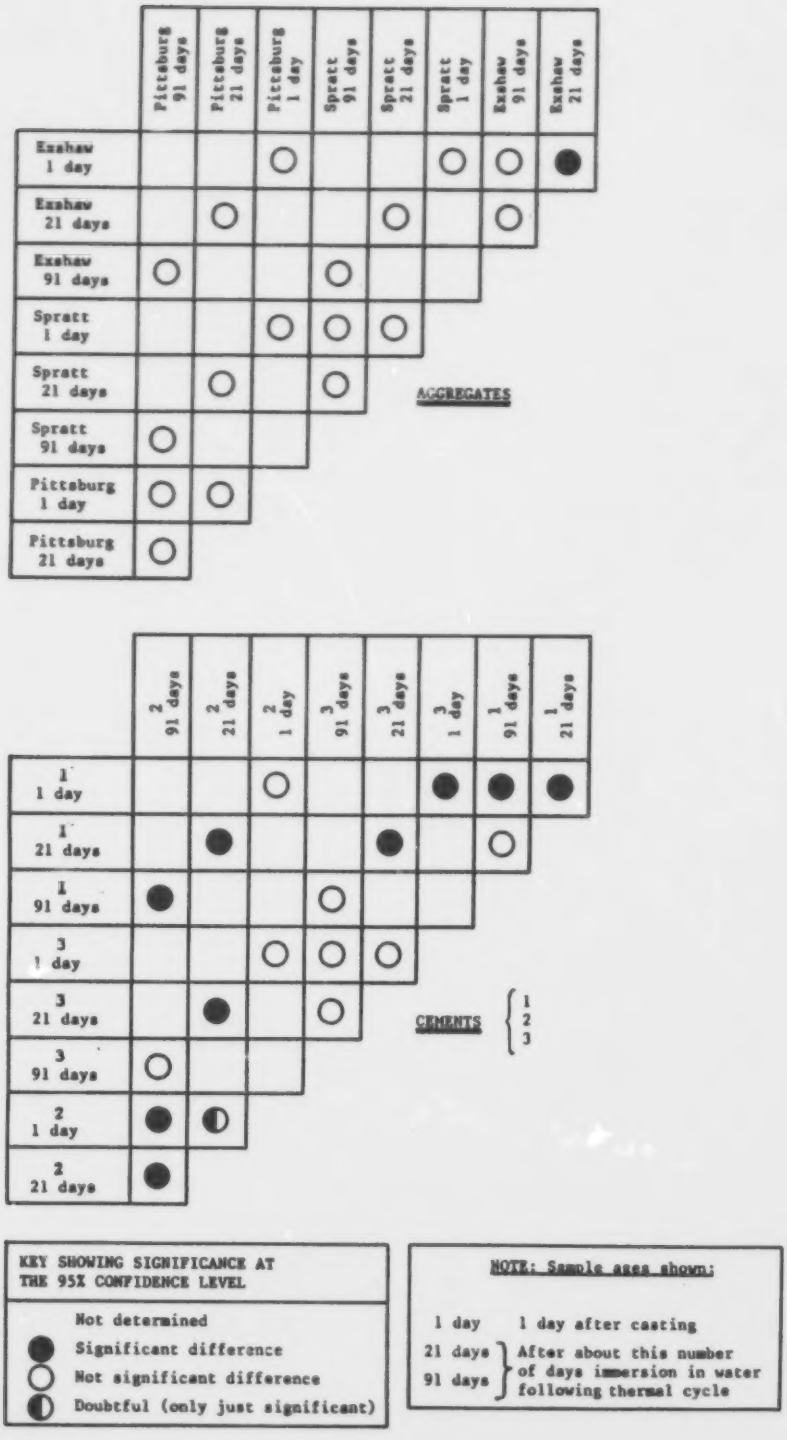

Fig.3. Results of Student's 't' significance tests on microcrack frequency statistics.

the growth of ettringite was probably facilitated by the Duggan-test. However, no apparent disruption of the cement paste surrounding the pockets of ettringite crystals was observed.

5. ANALYTICAL METHODS

5.1 Infrared Spectroscopy

Powdered specimens were prepared as KBr pellets and infrared spectra were recorded as percent transmission over two ranges of wave numbers from 250 to 1800 cm^{-1} and 3000 - 3800 cm^{-1}. Spectra were recorded before and after heating the pellets at 105°C for 24 hours. Samples of each aggregate and cement and the nine control mixes after 24 hours accelerated curing were studied.

Unhydrated cement samples showed typical spectra of Portland cement with cement 1 containing less sulphate than cement 2 or 3. This accords with the chemical analyses (section 2.2) which showed a lower SO_3 content in cement 1. The IR spectrum of cement 3 showed a trace of hydrated lime, $Ca(OH)_2$, which was absent in the other cements.

Control samples showed very strong absorption due to carbonates present in the aggregates making it very difficult to detect minor phases in the cement paste, such as ettringite. The traces of alkali-silica gel observed microscopically in control samples with Spratt aggregate were also below the detection threshold. Hence further IR analyses will be conducted only when necessary to aid the identification of deposits in cracks and voids.

5.2 X-ray diffraction

X-ray diffractograms were run over the range 2 to 78°2θ using $Co_{K\alpha}$-radiation. Analyses were made of each aggregate type and each cement in the unhydrated and hydrated states. Specimens of each concrete mix were also analyzed at different periods before and after the Duggan-test, together with corresponding control samples.

Major phases detected in all three aggregates are: calcite, quartz and dolomite. Spratt and Exshaw limestones contain the highest proportion of calcite, whilst Pittsburg aggregate contains a higher proportion of dolomite. The most quartz was detected in Spratt, followed by Pittsburg and Exshaw. Exshaw aggregate contains only a trace of dolomite.

All three unhydrated cements show characteristic lines of the major cement minerals, i.e. C_3S, C_2S, C_3A and C_4AF. There are however some noticeable differences in the amounts of both major and minor phases. Cement 1 contains the lowest amount of C_3S and C_3A but the highest amount of C_4AF. Gypsum, small amounts of MgO and possibly CaO are also present. Cement 2 shows the highest amount of C_3S with slightly less C_4AF and more C_3A and gypsum than cement 1. Traces of MgO, but no CaO are present. Cement 3 contains the highest proportion of C_3A, MgO and CaO, intermediate C_3S and the lowest C_4AF. The

greatest amount of SO_3 is present as anhydrite possibly accompanied by hemihydrate and a trace of gypsum.

All three cement pastes after 24 hours accelerated curing show very similar spectra consisting of CH, some unhydrated phases, small amounts of C-S-H and C-A-H and traces of calcite and ettringite. The same phases with a higher ratio of hydrated to unhydrated products are detected after 20 and 90 days curing.

The relative proportions of ettringite and portlandite (CH) detected in control and after-test samples are listed in Table 2. Ettringite is absent in all control samples after 1 day accelerated curing; traces of this mineral appear in most samples cured for over 90 days in 100% R.H. at both ambient temperature and 38°C. Ettringite in different quantities is present in all specimens immersed in distilled water after the Duggan-test. The largest amount occurs in mixes made with cement 2 (the amount decreasing with respect to aggregate type in the order Pittsburg > Spratt > Exshaw) followed by mixes with cements 3 and 1. Concretes made from cements 3 and 1 with each aggregate contain relatively small quantities of ettringite. Therefore differences in the proportions of the mineral in these concretes are less pronounced

Table 2: Relative proportions of ettringite and portlandite detected by X-ray diffraction in cement pastes

Sample Age	Phase	Ett	CH	Ett	CH	Ett	CH
	Mix:	cement 1/Exshaw		cement 1/Pittsburg		cement 1/Spratt	
1d	control	0	7.0	0	> 10.0	0	8.0
90d	control	0.5	8.0	0.5	9.5	0.5	10.0
90d	38°C	0	9.0	0.5	10.0	0	7.5
90d	after test	1.0	> 10.0	1.0	10.0	0.5	7.0
		cement 3/Exshaw		cement 3/Pittsburg		cement 3/Spratt	
1d	control	0	> 10.0	0	10.0	0	10.0
90d	control 22°C	0	7.5	0	9.0	1.0	8.5
90d	38°C	0	7.0	0	8.0	0.5	8.0
90d	after test	1.0	> 10.0	2	7.0	2.0	9.0
		cement 2/Exshaw		cement 2/Pittsburg		cement 2/Spratt	
1d	control	0	9.5	0	10.0	0	9.0
90d	control 22°C	0.5	9.0	0.5	8.5	0.5	10.0
90d	38°C	0.5	9.0	0.5	10.0	0.5	7.0
90d	after test	2.0	9.0	3.5	10.0	3.0	7.0
						cement 1/Spratt alkali boosted	
1d	control					0	8.5
90d	control 22°C					0	7.0
90d	38°C					0.5	7.0
90d	after test					1.0	8.0

than in the concretes made with cement 2. Changes in relative proportions of portlandite (CH) are very small and do not show any specific trend.

5.3 Discussion

Formation of ettringite in hardened cement paste has long been considered to cause expansion and decrease in strength [2]. Heinz and Ludwig [1] have shown that accelerated curing at temperatures >75°C at 100% R.H. leads to delayed expansion, and this was attributed to the formation of secondary ettringite. The amount of expansion and proportion of ettringite formed was found to increase at higher molar ratios of SO_3 to Al_2O_3 and initial curing temperature.

It appears possible that the heating (to 80°C) and cooling cycle in the Duggan-test facilitates the secondary formation of ettringite during subsequent immersion in water. X-ray diffraction data and observations on the SEM suggest a correlation between the amount of ettringite and the magnitude of expansion in the Duggan-test.

6. SUMMARY AND DISCUSSION

6.1 General remarks

Dimensional change data obtained in the Duggan-test differ significantly from those obtained in the CSA test (A23.2 - 14A); results of the latter test confirmed known alkali-reactivity patterns. AAR therefore has been ruled out as a major cause of expansion in the Duggan-test certainly during the first 21 days of immersion in water. However, AAR may contribute to the expansion at later ages.

In the Duggan-test the rate of concrete expansion depended on cement type, and was in the order: cement 2 > cement 3 > cement 1, regardless of aggregate type. These results do not correlate either with alkali content of the cement or with the alkali-expansivity of the aggregates. It is therefore concluded that the nature of the cement itself is of critical importance. Normative C_3S:C_2S ratio, soundness (i.e. content of CaO, MgO and SO_3), strength and coefficient of thermal expansion may have to be considered to find the mechanism responsible for expansion in the Duggan-test.

The Duggan-test increased microcrack frequency in concretes containing cement 2 and 1. Concretes made with cement 3 showed unusually high crack intensities even before the Duggan-test, and the after test values were not significantly greater than the initial values. Aggregate type had little or no effect, and final concrete microcrack frequency depended on cement type and was in the order: cement 2 > cement 3 > cement 1, approximately matching the order of expansivity in the Duggan-test.

The differences in rate of microcracking of the concretes made with cement 3 versus cements 2 and 1 may be partly due to differences in the mineralogical composition of the sulphates. The sulphate is

largely in the form of anhydrite in cement 3, and as gypsum in cements 2 and 1.

Concretes made with cement 3 contained less ettringite than concretes made with cement 2 even though cement 3 had the highest SO_3 content. Possibly the difference in sulphate mineralogy in the unhydrated cement was also a factor here. Cement 1 contained the least amount of SO_3 and correspondingly the ettringite content of these concretes was the smallest.

6.2 CONCLUSIONS BASED ON THE PRESENT TEST PROGRAMME

a. Alkali-aggregate reaction is not the major cause of expansion measured in the first 21 days of the Duggan-test.
b. The CSA concrete prism test (A23.2 - 14A) confirmed the known behaviour of expansive and non-expansive aggregates.
c. Expansion in the Duggan-test depends mainly on properties of the cement.
d. The amount and form of SO_3 in unhydrated cement may affect the initial and final frequency of microcracking induced by the Duggan-test.
e. Unusual behaviour of concretes made with cement 3 is possibly due to the presence of anhydrite rather than gypsum in the cement.
f. The amount of expansion in the Duggan-test at ages up to 90 days correlates with the relative proportion of ettringite and frequency of microcracking in the concrete samples.

REFERENCES

1. Heinz, D. and Ludwig, U., "Mechanism of Secondary Ettringite Formation in Mortars and Concretes Subjected Heat Treatment", Concrete Durability, American Concrete Institute, Special Publication 100, v.2, 2059-2071, 1987.

2. Mehta, P.K., "Mechanism of Expansion Associated with Ettringite Formation", Cement and Concrete Research, v.3, pp.1-6, 1973.

3. Rogers, C.A., "General Information on Standard Alkali-Reactive Aggregates from Ontario, Canada", Engineering Materials Office, Ministry of Transportation, Ontario, Canada, 1988.

4. Gillott, J.E., "Petrology of Dolomitic Limestones, Kingston, Ontario, Canada", Bulletin Geological Society of America , v.74, No. 6, pp.759-778, 1963.

5. Scott, J.F. and Duggan, C.R., "Potential New Test for Alkali-Aggregate Reactivity". Proceedings of the 7th International Conference on Alkali-Aggregate Reactions, Noyes, New Jersey, U.S.A., pp.319-323, 1987.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

MICROSTRUCTURAL ASPECTS OF A.A.R. : COMPARATIVE STUDIES OF LABORATORY AND FIELD SPECIMENS

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1. INTRODUCTION

This paper reports successively and comparatively analytical results on two kinds of samples developing an alkali silica reaction :

- identified deteriorated field specimens,
- laboratory made mortars and concretes.

The final objective is a better understanding of some microstructural aspects observed in real structures, through the precise study of the microstructure evolution, of laboratory made samples expected to develop the same type of reaction.

To achieve this objective, several conditions must be met in advance :

. characterization of the old field concrete, including the resulting expansive products, as well as the source of reactive silica, mainly by microscopic studies and chemical analysis (EDS).

. precise determination, on the laboratory made specimens, of the microstructure transformations, from the time of mixing ; characterization of the aggregates and detection of the first gels will be emphasized.

The idea beyond this study is that, while it is very difficult or even impossible to determine what has been the evolution of microstructure in field concretes because they are old (10 - 50 years), this can easily be done (if not easily explained) on laboratory made samples. From these a hypothesis can be formed to explain the sequence of deterioration in real structures.

In both types of specimens, the coarse aggregate is limestone containing a few percent of reactive silica :
The field concretes come from a road bridge near Quebec City ($\simeq$ 30 years old), while the laboratory samples have been made with Spratt's aggregate, according to preparation and

storing conditions described in our copending paper [1] and partly in our presentation at the Kyoto conference [2].
The microstructure observations will also be correlated with the expansion measurements.

2. ANALYTICAL TECHNIQUES. SAMPLE PREPARATION

2.1 Microscopy

The main technique employed for this study is scanning electron microscopy. Our results rely on a large series of micrographs, however, as the number of published photos cannot be too high, it will be limited here and they will all be shown during the oral session.

2.1.1 Optical microscopy has currently been in use. Thin sections are observed with a polarising transmission microscope (Axioplan Zeiss).

2.1.2. S.E.M. and energy dispersive analysis The S.E.M. microscope was the jeol 840 model. The samples under study are fresh fractures and polished sections. The former are metallized (Au/Pd) for morphological observation purposes. The latter, covered with a carbon film, are more adapted for quantitative chemical analysis with an energy dispersive spectrometer (Tracor, 524) ; two modes of observation can be selected : Secondary electrons (S E I) or back scattered electrons (B E I), which allows a quick but rough approximation of the chemistry of the solids.

X-ray mapping has also been conducted for precise localization of selected phases, especially the reactive silica in the Spratt aggregate.

2.2 Chemical and XRD analysis

have been used to complement some microstructural aspects in the Spratt aggregate investigations.

3. FIELD CASE STUDY

3.1 Presentation

We have had several opportunities to visit deteriorated structures in Canada and to get small pieces of concrete for laboratory investigations. This one, coming from the road bridge du Vallon - Charest near Quebec city, is approximately 30 years old. This structure has already been identified as being the seat of severe A.S.R., and field inspection has been reported [3] ; microstructural investigations have also been realized. [7, 8].

3.2 The Coarse Aggregate

After verification had been made that the sand (granitic type) was not reactive, the coarse aggregate was studied : it is basically made of limestone, including fossil organisms, and contains different types of silica known to generate an alkali silica reaction : beautiful chalcedony, or very fine chalcedony, and rings of spongy silica are clearly detected with the optical microscope, but the fine and detailed mophology of the reactive silica is better revealed with the S.E.M., as shown in micrograph 1.

Figure 1 : Spongy reactive silica. A few fibrous reaction products, are also visible

Observe the small size ($\simeq$ 1 μm) of individual particles, and their peculiar arrangment.

The second type of reactive silica often encountered in these samples as well as in Spratts aggregate is presented in micrograph 2.

Figure 2 : Micromorphology of another type of reactive silica. Lamellar crystals are lying on the surface.

3.3 Reaction Products in Concrete

Both forms of reactive silica, and possibly others which may have fully reacted, have generated expansive silico alkaline products through the attack of a high OH^- level.

The samples are badly damaged : the reaction products are very abundant and found everywhere, however a preferential localization was observed : gels filling completely or

partly the air voids ; rosette like crystals within fine or large cracks in the aggregates. The exact situation is somewhat more complicated :

3.3.1 Products of fixed composition Several types of crystals commonly found in old field concretes and already documented, - Cf. M. Regourd publications, especially [4, 5, 6] - are simultaneously present within the cracks, mainly the rosette - like and lamellar crystals.

The fairly constant composition found for these typical morphologies confirm previous findings [7] :

SiO_2 = 72, CaO = 17, K_2O = 9.5, Na_2O = 1.5 (weight %, expressed on the basis of anhydrous product).

The high content of CaO, the relative proportions of K_2O and Na_2O are interesting points to be considered. The presence of these stable phases indicates, that the field specimen has already undergone a long process of deterioration, but nothing can be said about how : this is the main purpose of the following chapter.

3.3.2 Low order crystallinity or amorphous products A general aspect is presented in micrograph 3, where blocks of gels exhibit a severe dessication, and appear like dried mud.

Figure 3 : Overall view of a gel area. At the surface, the gels are structured. Blocks have been removed, the surface beneath is smooth.

When looking at higher magnification, the morphology and chemical composition vary within a short distance :
For example, when moving along a block (100 - 200 µm), we have seen fibrous products, associated in small or large quantities, honeycombs, botryoidal textures but the most striking features are expressed in micrograph 4 :

- dessicated blocks of gels are pulling away from the surface beneath, which appears smooth,
- gels are torn apart and delaminated.
- the morphology and composition of the gels are not homogeneous : the thickness of the gels is indeed made of successive layers ; they start to split apart, at a place where the morphology and composition resemble that of the external surfaces.

- in these layers, the content of calcium is much higher than in the internal layers, which appear more compact.

Figure 4 : Detail of previous micrograph : the layered gels are delaminated

This type of micrograph raises the problem of the role played by calcium in the development of expansive gels. This will become apparent also in the laboratory made samples (see next section).

4. STUDY OF THE SAMPLES MADE WITH SPRATT AGGREGATE

4.1 Mortar Bars and Concrete Prisms : Brief Description

The preparation, placing, and storage conditions of these samples are the purpose of our pending paper [1], (refer to it for general information). The only points that will be mentioned here, are those more specifically dealing with the microstructure study :

For the expansion measurements, 4 specimens are required. A fifth one has been prepared from the same batch, for the microstructure study and kept in exactly the same conditions as the others.

In our opinion, this is an important point : we believe that it is only with similar samples in such identical conditions, that a correlation can be drawn, even though quantitative microstructure criteria cannot really be expected. Very few studies have been conducted in such a way, and rare, are the kinetic studies.

We should quote, as an example, that a comparative microstructure and composition study on conventional standard and new accelerated testing specimens, has been published by Shayan and Quick at the Kyoto conference [10].

4.2 Spratt's Aggregate

This can be considered as a reference (and a school) aggregate, representative of the alkali silica reaction. Similar to the previous case, this aggregate coming from a quarry near Ottawa is basically made of limestone. Recognized as being responsible of several deteriorations in structures, it is now available for testing through Ch. Rogers and the Ministry of Transportation of Ontario as well as a booklet containing useful data [9].

It's chemical and X.R.D. analysis bring a beginning of explanation , but the same analysis realized on it's insoluble residue are more informative (Table 1).

Table 1 : Chemical analysis of Spratt's aggregate and of its insoluble residue

	SiO2	Al2O3	Fe2O3	CaO	MgO	SO3	Na2O	K2O	L.O.I	TOTAL
SPRATT	8.9	0.6	0.3	48.85	1.45	0.30	0.03	0.09	39.7	100.2
SPRATT Ins	86.5	4.2	1.45	1.50	0	3.15	0.10	0.56	5.00	99.3

Besides limestone, the main constituant, the aggregate contains a few % of silica, and siliceous matter (mainly illite) ; some dolomite and pyrite were also found as traces.

Although this is not the scope of the study, it may be interesting to mention that, practising a modified version of the ASTM C-289 chemical method on the insoluble residue, clearly shows this aggregate as reactive (current research program).

4.2.1 Optical microscopy observations Several petrographers have observed thin sections of this aggregate : they all found the presence of some reactive silica (chalcedony, opal ?), but generally in lower proportion (1 - 2 %) than the real value determined by chemical analysis. This discrepancy will be explained in the following section.

4.3 Kinetic Study on Concrete Samples

It is clear that, if sensitive modifications related to ASR can be detected at early ages (1 or 2 months)by SEM, this would be a real advantage of the microstructure studies over conventional mortar bars and concrete prisms expansion tests, whose earliest results are at 6 months and rather at 12 months.

4.3.1 The study of 28 days specimens gives a clear picture of Spratt aggregate : micrograph 5. (BEI). Consider the following points :

1) Repartition of dolomite (dark grey spots),

2) The distribution and, more important, the quantity and size of the silica particles (they are more accurately observed in micrograph 6 at greater enlargment). The submicro silica occurs as isolated or slightly agglomarated particles, however the size of the nodules is generally below 10 µm. This fact plus the amorphous nature of the silica explain why it's content can be underestimated by optical microscopy.

3) The aggregate is porous and contains originally very fine pre-cracks, which will become privileged places for the penetration of the interstitial liquid phase.

Figure 5 : On the left, a Spratt aggregate. (polished section, BEI) : the light grey matrix is limestone. The dark grey, usually well outlined crystals are dolomite, (see also on the right). The small dark grey spots are silica or clays. Observe also the porosity, the fine cracks, and the etched edges.

Figure 6 : Enlargment of previous micrograph showing the micrometric size of the reactive silica (black).

The combining of points 2 and 3 establish favorable conditions to initiate the hydroxyl attack, and the formation of the initial gels. A clear demonstration is made in the next micrograph. We did not find any gels in the 28 days specimens (expansion is only 88 µm for the concrete containing 410 kg/m3 cement), but they became detectable in the

4.3.2 Two months old samples (G = 470 µm), as shown in Figure 7.

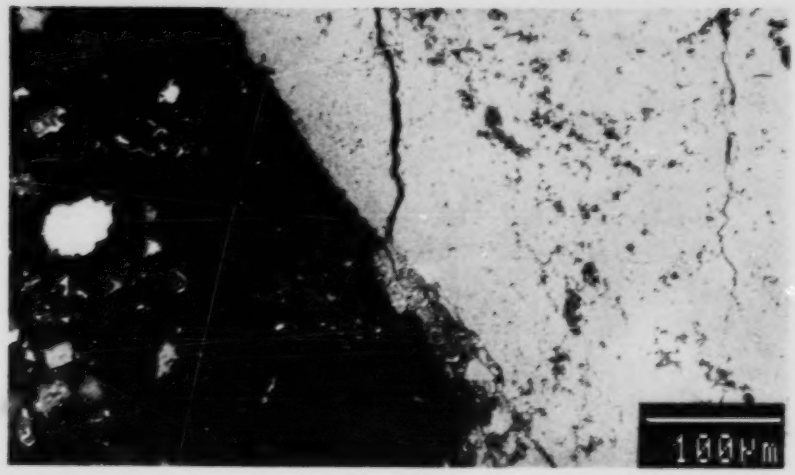

Figure 7 : Initial expansive gels in a 2 months old concrete located along a wide crack within an aggregate, and near an area containing reactive silica : top and bottom of the aggregate (grainy area). Note also that this grainy materials is pushed away from aggregate.

It is not possible to increase considerably the number of published micrographs, but the results of this section are supported by a series of pictures which will be discussed at the oral session.

Consider the crack on the left side and firstly, the top of it : it passes through a siliceous zone. Gels have not been identified in this area, which is the farthest from the mouth of the crack but somewhat further down, where they appear grainy. Small rounded protusions appear also, at their surface, essembling opal.

The EDS analysis on 3 points (A.C., Table 2), although not quantitative (gels are thin, so the analyser is taking into account surrounding phases, mainly the limestone matrix), are nevertheless instructive, if compared with the analysis of the next series of points located near the aggregate/paste interface (D.E.F.) :

Table 2 : EDS analysis of two series of gels (oxides, weight %)

Point	SiO2	K2O	Na2O	CaO	Al2O3	Fe2O3	SO3
A	35.95	3.19	2.57	51.05	2.61	2.78	0.98
B	37.23	2.66	1.76	51.18	1.49	1.75	3.21
C	37.53	2.61	3.27	49.55	2.06	1.94	2.97
D	47.75	3.12	0.30	46.88	0	0	1.27
E	57.75	3.11	0	38.72	0	0	0
F	53.59	2.96	0	42.54	0	0.62	0

4.3.3 3 and 4 months old specimens The 3 months old concrete did not show much difference, from the gels point of view but the physical appearance of the aggregate changed :

the number and width of cracks increased and a lot of new fine cracks appeared.

The network of silica also became more apparent ; as this is not due to an increase in silica, it may be related to the penetration of OH^- : the beginning of alkaline attack would accentuate the presence of the silica.

In the 4 months old concrete (G = 650 µm) drastic changes occured, as shown in Figure 8

- within the aggregates : enlargment and separation of cracks containing the gels ;
- appearance of gels outside of the aggregate : a ribbon of dessicated gel goes along the aggregate.

Although the new products still look like homogeneous gels, their chemical composition - which is now more accurately analysed (large rings) - has undergone some changes (Table 3).
(some oxides contents being of no real significance are not reported here).

Points A and B are representative of the gels inside the aggregate, while points C through F indicate composition of the external gels :
Gels inside are silica rich. The most important cation is Ca ($\simeq$ 20 % CaO). K_2O is around 5 % and Na_2O around 2 %. Inside or outside the Al_2O_3 content is homogeneous (1 1.5 %).

Table 3 : Composition of the gels in 4 months old concrete

Point	SiO2	K2O	Na2O	CaO	Al2O3
A	70.21	5.24	1.96	19.37	1.55
B	68.69	5.08	1.73	23.16	1.10
C	62.95	11.38	5.55	17.82	1.49
D	59.89	12.21	4.24	21.77	1.21
E	56.21	11.57	5.91	23.92	1.03
F	57.38	8	3.50	30.15	0.74

External gels contain much more cations : K and Na, while the SiO_2 content is depressed. The trend for CaO is not clear here. Gels are firstly formed within the aggregates in the vicinity of reactive silica : it is understandable that their SiO_2 content is high. The high level of calcium is not so easy to explain. We think that the initial gels contain high amounts of lime, and that they do not get enriched as a function of time. It remains however possible, that in real field specimens, this is not so and that gels get enriched through the process of portlandite attack, much later than in the laboratory made samples.

Other striking phenomena have been detected in these samples, but will be discussed during oral presentation.

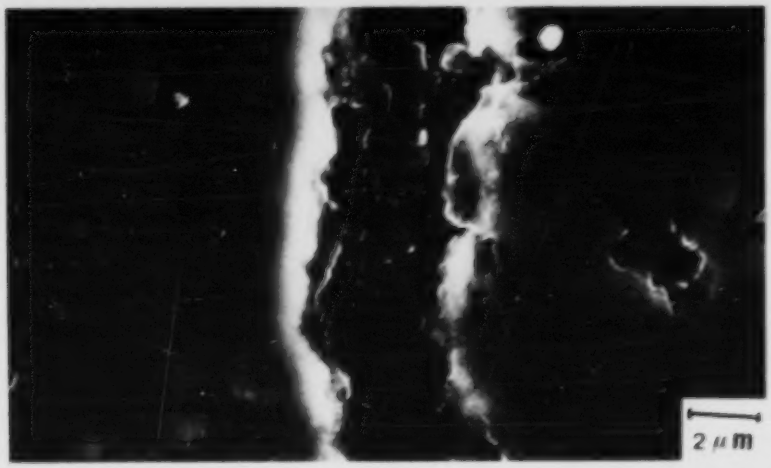

Figure 8 : Detail of previous micrograph : microstructure of the initial gels within a crack, repartition and microstructure of reactive silica (in small holes)

4.4 Mortar Bars

Common results with the concrete prisms will not be repeated, just the new ones will be given.

In the case of alkali silica reaction, mortars expand faster than concretes, therefore and as expected, at equivalent or superior aging times, mortars were more damaged than concretes.

4.4.1 3 months old mortar (G = 0.16 %) First of all, a dense network of cracks is observed within the aggregates ; large cracks are often empty, narrow ones may be partially filled. In these cracks typical composition of gels, which look homogeneous, is as given by points A, B, and C, Table 4 :

Table 4 : Composition of gels in 3, 4 and 10 months old mortars

Point	SiO2	K2O	Na2O	CaO	Al2O3
A	63.73	6.92	3.29	24.95	
B	66.90	5.81	3.71	21.97	
C	69.42	5.26	3.78	21.45	
D	67.25	9.28	5.24	16.34	1.47
E	67.34	9.76	4.91	16.66	0.88

As already mentioned, the silica content decreases from the inner to the outer gels, whereas the lime and potassium contents increase.

4.4.2 The 4 months old mortar (G = 0.23 %) In parallel with large internal cracks, where points D and E (Table 4) have been taken, gels were often found at the paste aggregate interface. The results exhibit the same trend as that observed for both series of points

reported in Table 3 : gels at the paste aggregate interface contain much more potassium and sodium than those inside. In this case, the CaO content is lower for the external gels.

4.4.3 The 10 and 12 months old mortars (G = 0.38 and 0.40 %) The samples are badly damaged ; some cracks are several tens of micron wide.
The gels have deeply changed : they commonly appear in the form of large ribbon within the aggregates, and their composition varies along the thickness of the gel (micrograph 9).

This is confirmed by the observations on fractures : gels start to exhibit morphological transformations, and a very wide variety has been observed, eg : botryods, microreniforms, no crystalline phases have been detected, but some structured gels have a similar chemical composition.

New fresh gels are also formed, and again, the determined CaO content is extremely high (40 - 50 %).

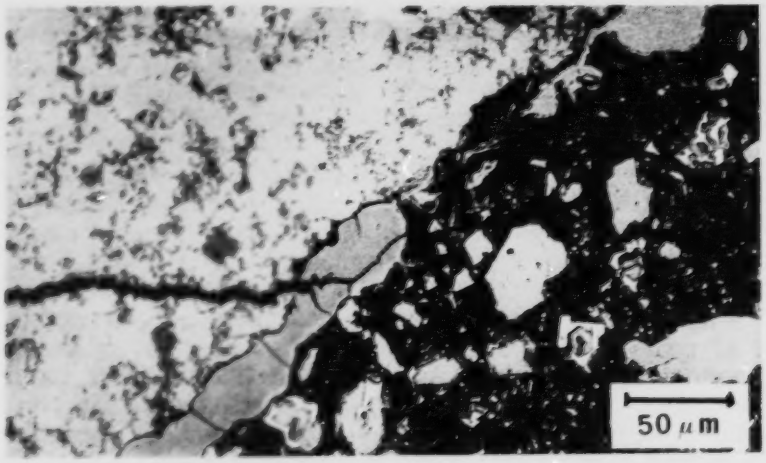

Figure 9 : Development of large ribbons of gels outside of the aggregate. A large crack within the aggregate is partly filled with gels. The reactive silica is easily observable.

5. DISCUSSION AND CONCLUSIONS

The SEM and analytical study of a series of young specimens made with the alkali silica reactive Spratt aggregate, provide relevant information on the modifications of microstructure leading progressively to the appearance of an intense network of cracks and to the formation of expansive gels and crystals of basic silicates.

5.1 The Aggregates Peculiarities

First of all, the detailed observation by SEM of the aggregate itself is essential with this type of aggregate. The classical observations by polarizing microscopy may fail to detect the real content of silica (amorphous and small size) ; even when agglomerated, the size remains

small (~10 µm).

The situation is somewhat different with the coarse aggregate in the field concrete : large rings of spongy silica and nice crystals of calcedony are easily detectable by optical microscopy.
Both these examples show that care must be taken when studying the petrography of limestone aggregates.

The SEM study of the Spratt aggregate revealed another important feature : the existence of very narrow linear pre-cracks. When they pass through the silica islands, this becomes a determining factor which favors the initial penetration of highly alkaline liquid phase. This is really the starting point of the deterioration process.

5.2 The Initial Gels

Alkali silica gels have been detected, at very early ages (2 months, G = 0.047 %), in small cracks passing through reactive silica : they look viscous and homogeneous. Their composition, even though not quantitatively determined, shows the main constituant is silica, associated with a few % of potassium and less sodium. The exact content of calcium is not known, owing to the interference of the limestone matrix, but it is almost sure that a high quantity of lime enters the gels, from the beginning.

Within a short time (1 or 2 months), the gels are localized at the same places, but their morphology starts to become modified : the surface, which now appears grainy, look like a precursor of the honeycomb morphology. Gels are later found in new places, especially along the paste aggregate interface. Their composition differs from that of the internal ones.

A few months later, the gels become structured, and new, characteristic morphologies, such as honeycombs and botryoidal textures are observed, on fractured specimens, whereas observation of polished sections in the backscattered election mode and subsequent EDS analysis confirm prevous findings : the gels are not homogeneous any more ; they are made of several layers, appearing more or less compact. The compact layers contain the highest silica %, the more porous ones (botryoidal textures for example) are highly calcic.

It has also been observed that the layers of gels tend to get separated precisely where the porosity and the lime content are high.

REFERENCES

[1] Ranc R., Cariou B., Sorrentino D., Storage conditions and reliability of the ASTM C227 mortar bar and CSA A.23.2 - 14 A concrete prisms tests : another look ; Ministry of Tranportation, Engineering Materials Report 92 , March , 1990.

[2] Sorrentino D., Ranc R., Cariou B., Methodology of an industrial research laboratory to assess the reactivity of aggregates. Focus on reproductibility problems, 307, 8th ICAAR, Kyoto, 1989.

[3] GREGI, cahier 3 : Atelier et visite, cours GGL-AO487, Université Laval, mai 1988.

[5] Regourd M., Hornain H., Microstructure of reaction products, 7 th ICAAR, p 375, Ottawa, 1986.

[6] Regourd-Moranville M., Products of reaction and petrographic examination, p 445 - 455, 8 th ICAAR, Kyoto, 1989.

[7] Berubé M.A., Fournier B., Les produits de la réaction alcalis - silice dans le béton : étude de cas de la région de Québec. 24, 271 - 288, Canadian Mineralogist, 1986.

[8] Ranc R., Sorrentino D., Bayle A., Un exemple de réaction alcalis granulats : l'échangeur routier du Vallon - Charest, (Québec). Rapport interne L.C.R., 1989.

[9] Rogers C.A., Engineering Materials Office, Ministry of Transportation general information on standard alkali-reactive aggregates from Ontario, 1988.

[10] Shayan A., Quick G., Microstructure and composition of AAR products in conventional standardand new accelerated testing, 475 - 482, 8 th ICAAR, Kyoto, 1989.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

CONCRETE PRISM EXPANSION TESTING TO EVALUATE SLOW/LATE EXPANDING ALKALI-SILICATE/SILICA REACTIVE AGGREGATES

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ABSTRACT

A group of known deleteriously reactive aggregates were tested in the concrete prism expansion test. The slow/late expanding aggregates did not cause significant expansion at 23° or 38°C when the concrete alkali level was 5.1 kg/m^3 Na^2O equivalent or less. Known alkali-silica reactive aggregates did cause significant expansion under these conditions. The slow/late expanding aggregates did cause significant expansion at 38°C when concrete alkali levels were increased to about 9 kg/m^3 Na_2O equivalent. Known non-reactive aggregates did not cause significant expansion under these conditions.

INTRODUCTION

The 1986 revision to the Canadian Standards Association [1] standard for concrete materials (Appendix B, CAN3-A23.1) recognized three types of alkali-aggregate reaction:

alkali-silica reaction;
slow/late expanding alkali-silicate/silica reaction;
alkali-carbonate reaction

The mechanisms of these expansive reactions are still not clearly understood. The alkali-silica reaction (ASR) is associated with the dissolution of reactive silica and the formation of alkali-silica gel in concrete. The slow/late expanding alkali-silicate/silica reaction (SLEASS reaction) is associated with the expansion of coarse aggregate particles in addition to gel formation. The alkali-carbonate reaction is associated with the expansion of coarse aggregate particles, not with dissolution of silica or formation of alkali-silica gel.

The SLEASS reaction is distinguished from ASR by different responses to accelerated test procedures as well as by different petrographic characteristics. Normally concrete affected by SLEASS reactions does

not show cracking until many years after construction, typically more than 10 years. The amount of alkali-silica gel present in the deteriorated concrete is usually much less than in concrete affected by ASR. The aggregates found to be susceptible to SLEASS reaction are greywacke, argillite, quartzwacke, quartzite, phyllite, arkose, sandstone and granite with highly strained quartz. The age of these rocks ranges from middle Precambrian in Ontario to Silurian in New Brunswick. These aggregates typically give relatively low amounts of expansion in mortar bar expansion tests compared with alkali-silica reactive aggregates. Normally mortar bar expansion is less than 0.1% at an age of one year using 1.25% Na_2O equivalent alkalies. Alkali-silica reactive aggregates usually give expansions considerably greater than 0.1% at six months under the same conditions. In concrete prism expansion tests at 38°C over water, the alkali-silica reactive aggregates give significant expansion when the alkali level of the concrete is greater than about 3.8 kg/m^3 of Na_2O equivalent. Figure 1 shows concrete prism expansion after one year against concrete alkali level for Spratts aggregate, an alkali-silica reactive, chalcedony bearing limestone. Figure 1 also shows the concrete prism expansion against concrete alkalies for Sudbury aggregate, a typical SLEASS reactive aggregate. Greater amounts of alkali (5 kg/m^3 Na_2O) are needed, compared to ASR aggregates to induce significant expansion and cracking under laboratory conditions.

In Ontario, the SLEASS reactive aggregates are recognized by petrographic examination. A source of coarse aggregate is not be used in concrete for highway use if it contains more than 15% of the potentially reactive aggregate types, unless field performance or results of accelerated testing are acceptable. Typically those sources that have the worst field performance contain significantly more than 15% reactive particles, values of between 40 to 80% are typical (Magni, Rogers and Grattan-Bellew [2]. The value of 15% was chosen to provide a satisfactory margin of safety.

For the past six years, the Ontario Ministry of Transportation has been conducting extensive concrete testing in an effort to induce cracking in the laboratory and to confirm the adverse field performance of these aggregates. This paper describes the results of this testing.

SAMPLE COLLECTION

Fourteen coarse aggregates from Ontario were tested in this study. The location of these aggregates is shown in Figure 2 and field performance in Table 1. Three aggregates from Quebec and New Brunswick were also tested. The Nelson aggregate from Burlington, Ontario was used as a known non-reactive laboratory control with a natural fine aggregate from Guelph, Ontario. The Guelph fine aggregate was also used as the fine aggregate in most of the mixtures with the other aggregates. The Spratts (Ontario) and Beauceville (Quebec) aggregates were included as typical alkali-silica reactive coarse aggregates for comparison with the SLEASS reactive group. The Rok Engineering Quarry

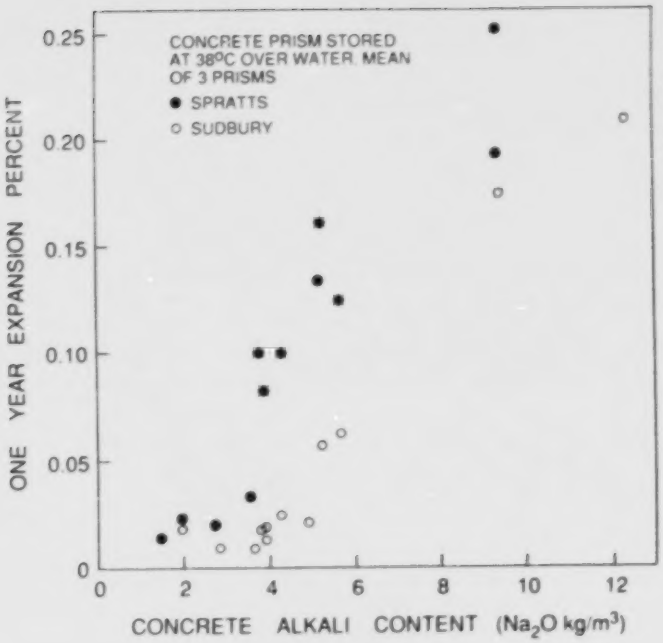

FIGURE 1.
Concrete prism expansion against concrete alkali content for Spratts and Sudbury aggregates.

FIGURE 2 - Location of Ontario aggregates tested in this study.

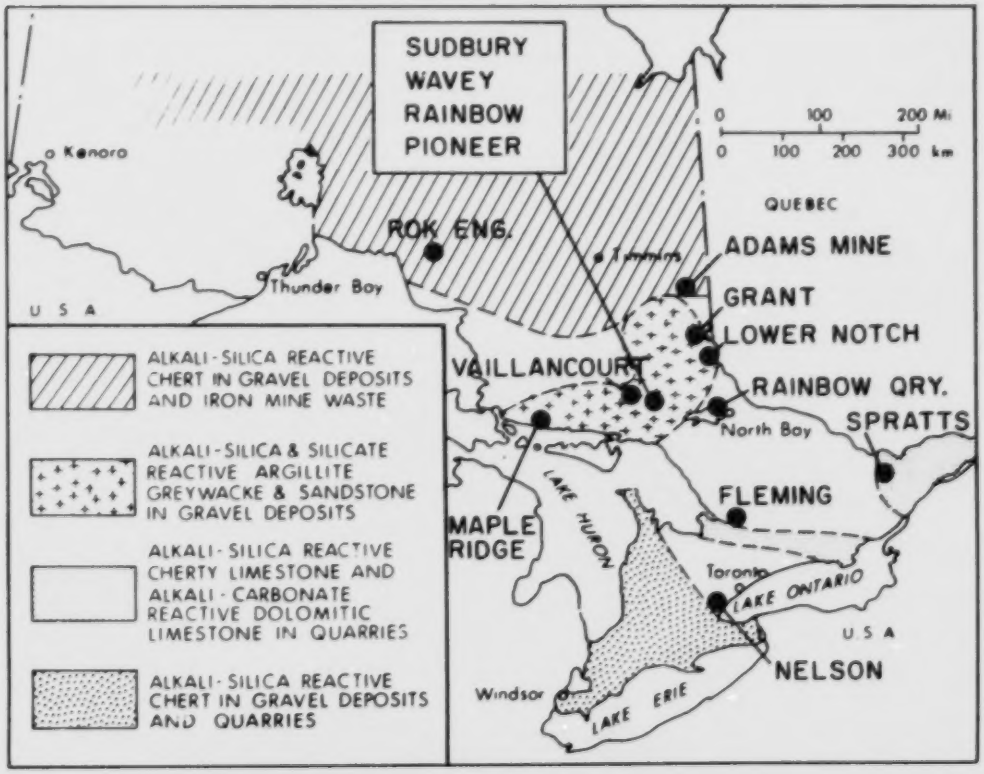

TABLE 1 - Coarse Aggregate Location and Performance Data

Aggregate Sources	Location	Remarks
Fleming	Washago, Ontario	Quarried granite with strained quartz petrographically similar to granite used in 70 year old reactive conrete.
Maple Ridge	Thessalon, Ontario	Huronian Supergroup gravel used in several bridges 1957/8, reactive.
Sudbury	Sudbury Airport, Ontario	Huronian Supergroup gravel used in many bridges, 1950's onwards, reactive.
McNulty	St. John, New Brunswick	Beach gravel used in several structures in St. John, reactive.
Adams Mine	Kirkland Lake, Ontario	Quarried cherty iron formation used in 1969 bridge, reactive.
Lower Notch	Cobalt, Ontario	Quarried Huronian Supergroup argillite used in Lower Notch Dam with 20% flyash, reactive.
Springhill	Fredericton, New Brunswick	Quarried metamorphosed Silurian sediments, used in Princess Margaret bridge, reactive.
Grant	New Liskeard, Ontario	Huronian Supergroup gravel used in 1969 bridge and others, reactive.
Pioneer	Sudbury, Ontario	Huronian Supergroup gravel typical of that used in many bridges, reactive.
Rainbow Gravel	Sudbury Airport, Ontario	Huronian Supergroup gravel typical of that used in many bridges, reactive.
Rok Engineering	Hemlo, Ontario	Quarried Archean granite used in Hemlo mine development, non-reactive.
Vaillancourt	Dowling, Ontario	Huronian Supergroup gravel with low % reactive components, reactive.
Wavey Gravel	Sudbury Airport, Ontario	Huronian Supergroup gravel typical of that used in many bridges, reactive.
Rainbow Quarry	North Bay, Ontario	Quarried Grenville gneiss with trace of strained quartz, non-reactive.
Spratts Quarry	Stittsville, Ontario	Quarried Bobcaygeon Formation cherty limestone, alkali-silica reactive.
Beauceville	Beauceville, Quebec	Quarried rhyolite, used in Sartigan Dam, alkali-silica reactive.
Nelson	Burlington, Ontario	Quarried Silurian dolostone, non-reactive.

and Rainbow Quarry aggregates were not considered to be deleteriously alkali reactive but both lack long term field performance in highway structures. The remaining aggregate sources were considered to be SLEASS reactive aggregates. The quality of field performance information is variable, some having well documented poor field performance (Maple Ridge, Sudbury, Adams Mine, Rainbow Gravel, Wavey Gravel). Others had less well documented field performance (Fleming, McNulty, Lower Notch, Springhill, Grant, Pioneer, Vaillancourt).

LABORATORY TESTING

The aggregates were oven dried, graded and subjected to conventional aggregate physical durability tests and petrographic examination. The results are shown in Table 2.

A number of different concrete mixture designs were used in this study:

1 310 kg/m^3 of cement with about 0.9% Na_2O equivalent alkalies. The alkali level was increased to 1.25% Na_2O by the addition of NaOH to the mixing water. These concretes had a calculated alkali content of 3.9 kg/m^3 of Na_2O equivalent alkalies.

2 415 kg/m^2 of cement with 1.17% Na_2O equivalent alkalies. The alkali level was increased to 1.25% Na_2O by the addition of NaOH to the mixing water. These concretes had a calculated alkali content of between 4.9 and 5.1 kg/m^3 of Na_2O equivalent alkalies.

3 310 kg/m^3 of cement with about 0.8% Na_2O equivalent alkalies. The alkali level was increased to 3.0% Na_2O by the addition of NaOH to the mixing water. These concretes had a calculated alkali content of between 9.0 to 9.3 kg/m^3 of Na_2O equivalent alkalies.

A number of 75 x 75 x 400 mm concrete prisms were made from the various aggregate/mixture combinations. These prisms were made, cured demolded and length measured following the Canadian Standards procedure (CAN3-A23.2-14A, 1986). Following measurement for initial length, the prisms were stored in a number of different environments and periodically measured for change in length.

RESULTS AND DISCUSSION

Nearly all concrete prisms with alkali contents of 5.1 kg/m^3 Na_2O equivalent or less, stored at 23°C in the moist curing room, gave less than 0.024% expansion and usually less than 0.019% expansion at one year. The only exceptions were the alkali-silica reactive Spratts (0.087%) and Beauceville (0.037%) aggregates. More detailed data is not presented.

TABLE 2 - Coarse Aggregate Physical Test Data and Composition

Laboratory Test	Sample Data						
	Maple Ridge	Lower Notch	Adams Mine	Rainbow Quarry	Springhill Quarry	McNulty Gravel	Sudbury Gravel
Petrographic Number (H.L.)	100	105	134	127	182	108	117
Petrographic Number (Gran.)	100	100	100	102	108	100	100
Absorption, percent	0.50	0.37	0.27	0.53	0.50	0.50	0.46
Bulk Relative Density	2.74	2.68	3.05	2.67	2.72	2.71	2.71
Magnesium Sulphate Soundness, loss in percent	1	0	1	2	1	-	1
Los Angeles Abrasion, loss in percent	14	11	18	33	12	-	13
Petrographic Composition (percent)							
Non-Reactive Components							
Granite (Non-Strained)/Gabbro	47	5	11	22	0	53	25
Gneiss	2	0	7	78	0	8	0
Meta-Volcanic	0	2	2	0	0	12	0
Potentially Reactive Components							
Sandstone, Arkose	19	29	Taconite	0	0	12	32
Quartzite	4	0	(Cherty Iron	0	0	2	11
Greywacke, Argillite	28	64	Formation)	0	100	10	32
Strained Quartz	0	0	80%	Trace	0	Chert 3%	0
Total Potentially Reactive Components in Percent	51	93	80	Trace	100	27	75

TABLE 2/continued Coarse Aggregate Physical Test Data and Composition

Laboratory Test	Sample Data						
	FLEMING	ROK ENG	PIONEER	WAVEY	RAINBOW	GRANT	VAILLANCOURT
Petrographic Number (H.L.)	119	100	107	106	109	112	100
Petrographic Number (Gran.)	100	100	100	102	102	100	100
Absorption, percent	0.38	0.40	0.40	0.54	0.50	0.62	0.30
Bulk Relative Density	2.667	2.692	2.705	2.698	2.708	2.768	2.690
Magnesium Sulphate Soundness, loss in percent	3	2	1	1	2	1	0
Los Angeles Abrasion, loss in percent	49	30	15	14	16	13	17
Petrographic Composition (percent)							
Non-Reactive Components							
Granite (Non-Strained)/Gabbro	0	94	33	32	20	53	41
Gneiss	0	6	4	0	2	0	30
Meta-Volcanic	0	0	6	9	9	8	8
Pontentially Reactive Components							
Sandstone, Arkose	0	0	15	19	31	3	11
Quartzite	0	0	12	21	14	0	0
Greywacke, Argillite	0	0	30	19	24	36	10
Strained Granite	100	0	0	0	0	0	0
Total Potentially Reactive Components in Percent	100	0	57	59	69	39	21

Table 3 shows expansion at one year for concrete prisms stored at 38°C in closed steel boxes with filter paper wicks over water. The slow/late expanding alkali-silicate/silica (SLEASS) reactive aggregates generally did not show expansion greater than 0.04% at 38°C when mixed in concrete with about 5 kg/m^3 Na_2O equivalent alkalies. There were two exceptions; the Adams Mine and Springhill quarry aggregates. These gave concrete expansion of 0.071% and 0.097% respectively at 273 days. These aggregates are better classified as alkali-silica reactive since their mortar bar expansions exceed 0.1% at 6 months. If these two aggregates are excluded, the average expansion of the SLEASS group (n=6), for which data are available at both 5 and 9 kg/m^3 alkalies, was 0.025% at 5 kg/m^3, and 0.110% at about 9 kg/m^3 alkalies. An expansion level of less than 0.04% at one year would not normally be considered harmful according to the Canadian Standards Association maximum suggested expansion values (Table B1 of Appendix B, CAN3-A23.1, 1986). In contrast, the known alkali silica reactive Spratt and Beauceville aggregates gave 0.134% and 0.142% expansion respectively at about 5.0 kg/m^3 Na_2O equivalent alkalies (Table 3).

The expansion data (Table 3, Figures 3 and 4) for the SLEASS aggregates generally show significant expansion after one year when mixed in concrete with 9.0 to 9.3 kg/m^3 Na_2O equivalent alkalies. In contrast, the non-reactive aggregates (Rok Engineering quarry, Rainbow quarry and Nelson) showed low amounts of expansion at the artificially high alkali level of about 9 kg/m^3 of Na_2O equivalent. The highest expansion (0.044%) of this 'non-reactive' group was given by the Rainbow quarry aggregate. The Rainbow prisms were broken open and examined. They showed very small amounts of alkali-silica gel lining some air voids adjacent to quartz rich particles of gneiss. This aggregate is currently being used in Ontario highway work without any reported problems. It could be classed as 'very slightly reactive' but not deleteriously reactive.

Clearly, if it is desired to induce these known slow/late expanding alkali silicate/silica reactive aggregates to cause deleterious expansion and cracking of concrete in the laboratory, unnaturally high alkali levels are needed. Alkali levels of 9.0 kg/m^3 Na_2O equivalent would be unlikely to be attained in the field. The maximum alkali level found in an Ontario type 10 cement has been about 1.4% Na_2O equivalent. If this cement was used, a cement content of about 645 kg/m^3 would be needed to give a concrete alkali content of 9.0 k_b/m^3. The maximum cement content normally used in Ontario is about 475 kg/m^3 in some kinds of pre-cast work. Why are unnaturally high alkali levels required to cause significant expansion in the laboratory, yet in the field concrete made with these SLEASS reactive aggregates and alkali levels of about 4.0 kg/m^3 Na_2O cracks after about 10 years? The answer may lie in the phenomenon of excessive alkali leaching under laboratory conditions.

TABLE 3 - Concrete Prism Expansion Data at One Year

Aggregate Coarse	Source Fine	Alkali Content kg/m^3, Na_2O	One Year Concrete Expansion, 38°C, %	One Year Mortar Bar Expansion, %, Note 3
Fleming	Guelph[1]	3.9	0.014	0.033
	Guelph[1]	5.0	0.022	
	Guelph[1]	9.3	0.077*	
Maple Ridge	Guelph[1]	5.0	0.020	0.064
	Guelph[1]	9.0	0.040	
Sudbury	Guelph[1]	4.9	0.021	0.062
	Guelph[1]	9.4	0.174*	
McNulty	Guelph[1]	5.0	0.030	0.119
	Guelph[1]	9.2	0.129*	
Adams Mine	Guelph[1]	5.0	0.071[2]	0.158[4]
	Guelph[1]	9.2	0.110[2]	
Lower Notch	Guelph[1]	5.1	0.039[2]*	0.035
	Guelph[1]	9.2	0.100[2]*	
Springhill	Guelph[1]	5.1	0.097[2]*	0.312[4]
	Guelph[1]	9.2	0.206[2]*	
Grant	Grant[1]	3.9	0.006	0.058
	Guelph[1]	5.1	0.017	
	Grant[1]	9.1	0.138*	
Pioneer	Pioneer[1]	3.9	0.021	0.043
	Pioneer[1]	9.3	0.159*	
Rainbow Gravel	Rainbow[1]	3.9	0.023	0.045
	Rainbow[1]	9.3	0.240*	
Rok Engineering[1]	Guelph[1]	3.9	-0.002	-
	Guelph[1]	9.3	0.014	
Vaillancourt	Guelph[1]	3.9	0.007	0.025
	Guelph[1]	9.3	0.182*	
Wavey Gravel	Wavey[1]	3.9	0.013	0.053
	Wavey[1]	9.3	0.252*	
Rainbow Quarry[1]	Guelph[1]	3.9	0.008	0.032
	Guelph[1]	9.1	0.044	
Spratts	Guelph[1]	5.1	0.134*	0.319
	Guelph[1]	9.3	0.193*	
Beauceville	Guelph[1]	5.0	0.142*	0.337
	Guelph[1]	9.3	0.188*	
Non-Reactive Control				
Nelson[1]	Guelph[1]	3.9	0.010	Nelson
		5.0	0.015	0.036
Guelph[1]		5.2	0.014	Guelph
		9.2	0.027	0.048
		12.4	0.038	

NOTES: 1 - aggregate not considerd deleteriously reactive
2 - expansion data at 273 days
3 - bars with 1.25% Na_2O, in plastic sleeves
4 - results at 252 days
* - cracking observed

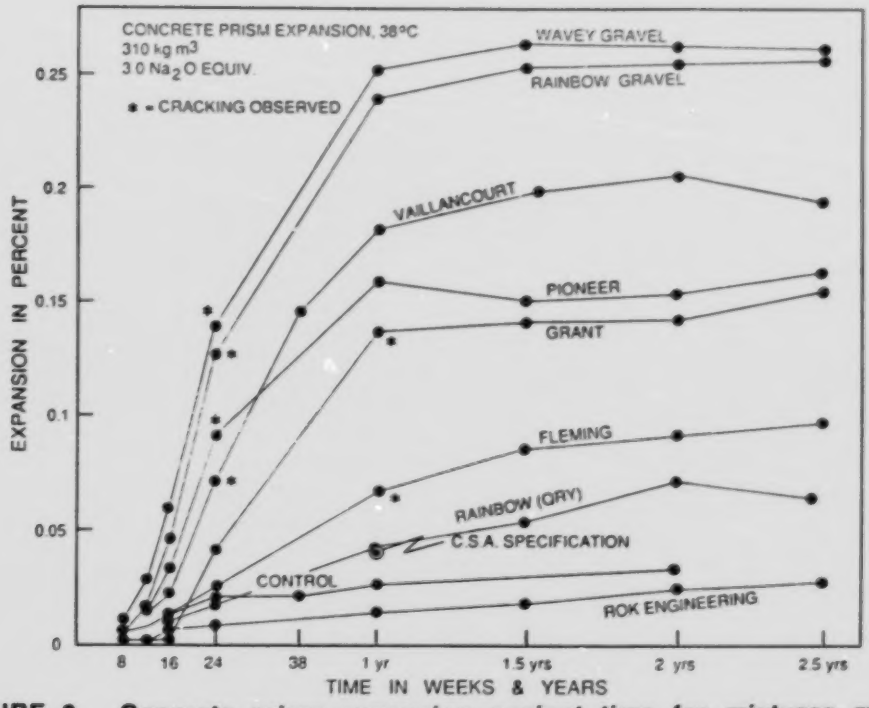

FIGURE 3 - Concrete prism expansion against time for mixtures made with 3.0% alkalies (about 9 kg/m^3), Group 1.

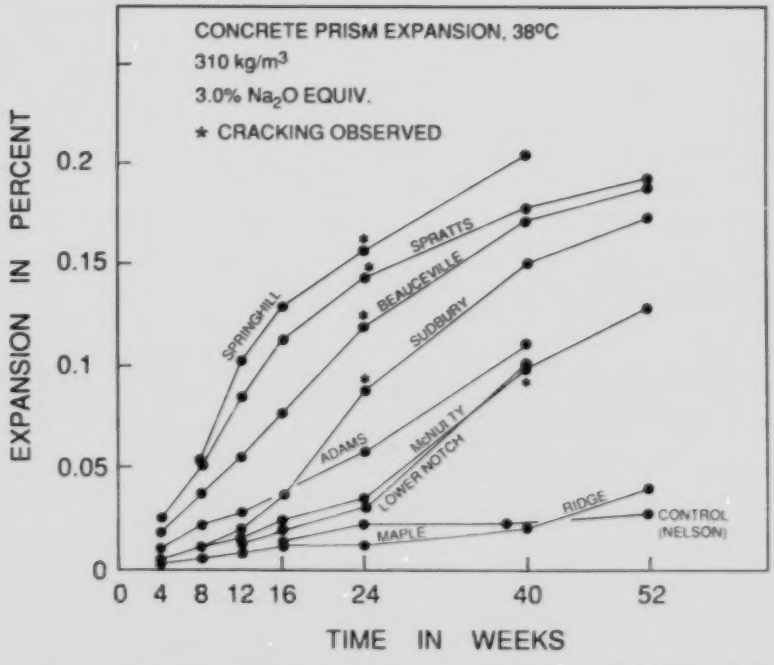

FIGURE 4 - Concrete prism expansion against time for mixtures made with 3.0% alkalies (about 9 kg/m^3), Group 2.

Rogers and Hooton [4] measured the amount of alkali remaining in concrete prisms, of the same size as used in this study, stored in a variety of conditions. Figure 5 is taken from the Rogers and Hooton paper and shows that between 63% and 22% of alkalies had been lost after 2½ years, depending on the storage condition. A concrete prism stored under identical conditions to that used in this study, in a steel box at 38°C, lost 42% of the original alkali after 2½ years. It is postulated that the alkali is removed by a process of condensation of water on the surface of the prism, dissolution of the very soluble alkalies, and dripping of the alkali laden condensed water into the water reservoir at the bottom of the container. It has been known for a long time that the water in the storage containers often contains large amounts of alkali (Blanks & Meissner, [5]). The removal of alkali slows down and eventually effectively stops alkali-aggregate reactions. In highway structures, such a leaching process must also take place but at a much slower rate. The dimensions of the members are an order of magnitude larger and the concrete is rarely exposed to continually moist conditions. The so called accelerated laboratory storage conditions of high temperature and continual moisture remove the alkali from the concrete rather quickly, and stop the reaction unless the initial alkali content was artificially increased to start with.

There are a number of possible ways of preventing this phenomenon:

1. Construct much larger concrete specimens for laboratory measurement so that alkali transport distance is increased and alkali leaching is thereby reduced. Bakker [6] found that the larger the cross section of concrete beams, the greater the expansion, confirming a similar observation by Davis [7];

2. Prevent leaching of alkalies by storing the concrete prisms in tight fitting plastic bags with excess water. In the Rogers and Hooton experiment this did not totally prevent alkali loss but only reduced it. British methods for concrete prisms, which call for wrapping the prisms in moist tissue and then placing in tightly fitting polythene sleeves [8] may be superior in this regard;

3. Prevent leaching by immersing the concrete in a neutral alkali balanced environment such as a sodium and potassium chloride solution. Eventually these alkalies will enter the concrete and result in an increase in alkalies (Figure 5). This may recreate some environments (highway bridges and parking garages) but is rather severe for other conditions;

4. Adopt the procedures used in this study; accept that leaching will take place and add sufficient additional alkali to nullify the effect of leaching on the progress of the reaction. High alkali levels (3%) have been used in studies of the alkali-carbonate reaction [9, 10]. High alkali levels have also been used by increasing cement contents to unnaturally high levels such as the 700 kg/m^3 in the draft British Standard Procedure [8].

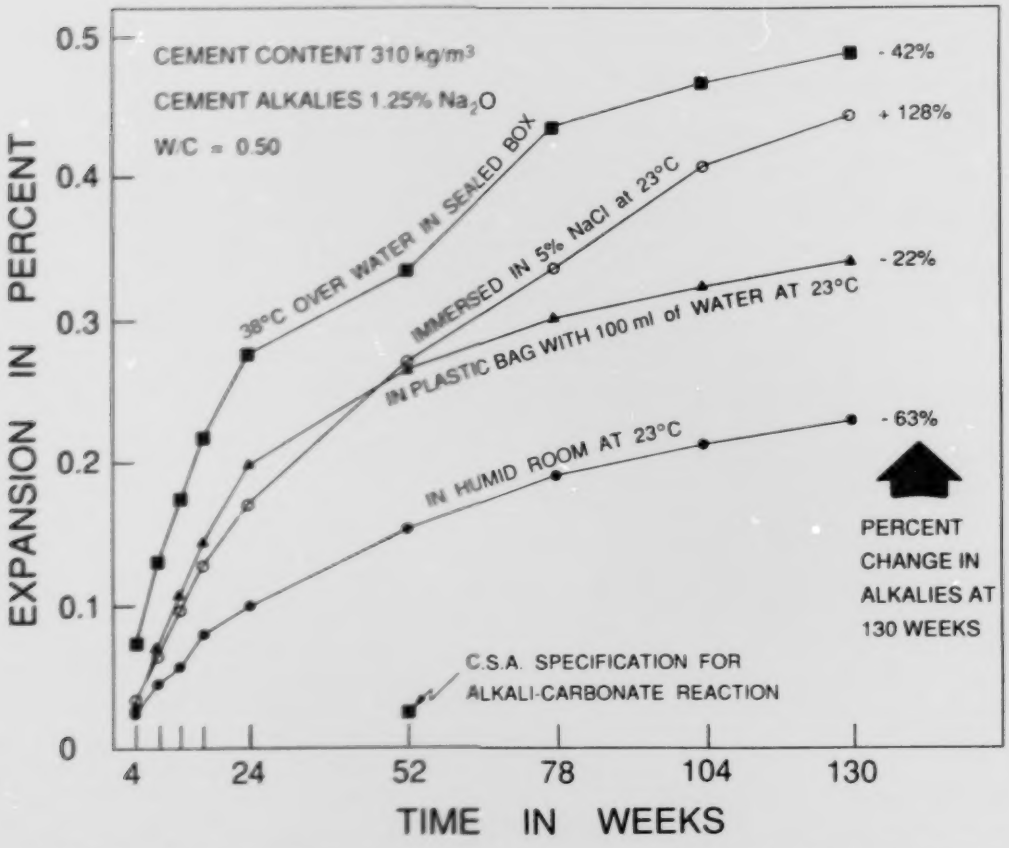

FIGURE 5 - Expansion of concrete prisms made with alkali-carbonate reactive coarse aggregate with change in water-soluble alkali content, from reference [4].

What are the purposes of measuring expansion of concrete in the laboratory? These tests are usually conducted for two reasons:

To determine whether or not the aggregate is potentially alkali reactive; and

To determine what are satisfactory control measures to prevent deleterious alkali reactive expansion taking place.

If testing is conducted to satisfy the first reason, identification of potentially reactive aggregates, there should be little objection to artificially increasing the alkali level of the concrete mixture. It may be argued that non-reactive aggregates may be made to appear reactive if such a procedure was adopted. The expansion of the Rok Engineering granite, the Rainbow gneiss and the Nelson dolostone (Figure 3) was low at the highest alkali level. A non-reactive aggregate cannot be made to appear reactive however high the alkali content. A marginally reactive aggregate, with good field performance, might give high expansion under these conditions.

If testing is performed to satisfy the need to know what corrective measures must be taken, artificially increasing the alkali content of the mixture is not satisfactory. Alkali leaching must be prevented, yet the speed of the reaction of the aggregate (if any) must be increased so that results may be available in a reasonable length of time. The concrete prism expansion test, as currently described, is probably not satisfactory for safely determining whether or not a proposed corrective measure is adequate.

What are useful test limit criteria to separate potentially reactive from non-reactive aggregates? Figures 3 and 4 are instructive. The Rainbow quarry aggregate, which has a good but limited performance record was judged not to be deleteriously reactive, gave 0.044% expansion at 1 year. The Maple Ridge aggregate gave an expansion of 0.040%. This aggregate sample was taken from a gravel source that was used in 1957 and 1958 to construct bridges in the Thessalon area. These bridges now show unmistakable signs of alkali reactivity damage which is judged to be deleterious. The other reactive aggregates tested in this study nearly all gave significantly greater expansion (0.077% or greater). The other non-reactive aggregates gave significantly less expansion (0.027% or less). The boundary between acceptable and unacceptable expansion, obtained under these testing conditions, must lie between the values 0.027% and 0.077%. The Maple Ridge and Rainbow quarry aggregates must lie in the gray area where it is most difficult to make a judgement especially when the wide interlaboratory variation of concrete prism expansion tests is considered [11].

The Canadian Standards suggested maximum expansion of 0.04% at 1 year is probably a reasonable value to separate potentially deleteriously reactive aggregates from non-reactive aggregates. Like all testing criteria it should not be regarded as an infallible number that separates good from bad.

CONCLUSIONS

Testing of a suite of known slow/late expanding alkali silicate/silica reactive rocks has led to the following conclusions:

1. These aggregates, when tested in concrete with 5.1 kg/m^3, or less, Na_2O equivalent alkalies, stored under moist conditions at 23°C gave one year expansions of less than 0.024%. Known alkali-silica reactive aggregates gave expansions of between 0.037% and 0.087% under the same conditions;

2. These aggregates, when tested in concrete with about 5 kg/m^3 Na_2O equivalent alkalies, stored under moist conditions at 38°C gave an average one year expansion of 0.025% with no expansion greater than 0.039%;

3. These aggregates, when tested in concrete at 38°C with about 9 kg/m^3 Na_2O equivalent alkalies gave one year expansions of between 0.040% and 0.252%. Three non-reactive aggregates gave expansions of between 0.014% and 0.044%;

4. The laboratory evaluation of the alkali reactivity of aggregates is difficult. At present there is no concrete prism test, at one alkali content and one temperature, that will identify all varieties of alkali reactive aggregate.

5. The current concrete prism expansion test at 38°C may not be effective in determining the efficiency of corrective measures to control alkali reaction. The test must be modified to prevent or compensate for alkali leaching which takes place under laboratory conditions;

6. A maximum linear expansion limit of 0.04% at one year separates the majority of non-reactive aggregates from those known to be deleteriously reactive under adverse exposure conditions.

REFERENCES

1. CANADIAN STANDARDS ASSOCIATION, 1986; Appendix B - Alkali-Aggregate Reaction; Canadian Standards Association, Rexdale, Ontario, Supplement No. 2 to CSA Standards CAN3-A23.1-M77 and CAN3-A23.2-M77, October, 37p.

2. MAGNI, E.R., ROGERS, C.A., and GRATTAN-BELLEW, P.E., 1987; The Influence of the Alkali-Silicate Reaction on Structures in the Vicinity of Sudbury, Ontario; Proceedings of the 7th International Conference on Alkali-Aggregate Reaction in Concrete, Ottawa, Canada, Noyes Publications, Park Ridge, N.J., U.S.A.; pp.17-22.

3. BERUBE, M.A., FOURNIER, B., 1988; Le Barrage Sartigan dans la Beauce (Quebec, Canada): Un cas-type de deterioration due beton par des reactions alcalis-granulates; Canadian Journal of Civil Engineering, V.14, pp.372-407.

4. ROGERS, C.A., R.D. HOOTON, 1989; Leaching of alkalies in alkali-aggregate reaction testing; 8th International Conference on Alkali- Aggregate Reaction, Proceedings, p.327-332, The Society of Materials Science, Kyoto 606, Japan.

5. BLANKS, R.F. and H.S. MEISSNER, 1946; The Expansion Test as a Measure of Alkali-Aggregate Reaction; American Concrete Institute Journal, 17, 5, 517-539, April.

6. BAKKER, R.F.M., 1983; The Influence of Test Specimen Dimension's on the Expansion of Alkali Reactive Aggregate in Concrete; Proceedings of the 6th International Conference on Alkalies in Concrete, Edited by G.M. Idorn, Steen Rostam, Danish Concrete Association, Copenhagen, pp.369-375.

7. DAVIS, C.E.S., 1957; Comparison of the expansion of mortar and concrete; Australian Journal of Applied Science, V.8, pp.224-234.

8. HOBBS, D.W.; Alkali-silica reaction in Concrete; Thomas Telford Ltd., London, 1988.

9. SMITH, P., 1974; 15 Years of Living at Kingston with a Reactive Carbonate Rock; Transportation Research Record 525, pp.23-27.

10. ROGERS, C.A., 1986; Evaluation of the Potential for Expansion and Cracking of Concrete due to the Alkali-Carbonate Reaction; Cement, Concrete and Aggregates, CCAGDP, V.8, No.1, pp.13-23, Summer.

11. ROGERS, C.A., 1990; Interlaboratory Study of the Concrete Prism Expansion Test; Ministry of Transportation, Ontario, Engineering Materials Office, EM Report 92.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

RAPID TEST FOR PREDICTING ALKALI REACTIVITY: PROMISES AND PROBLEMS

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1. ABSTRACT

Because of inherent slowness of Alkali - Aggregate Reaction (AAR) both in nature and in existing ASTM and CSA tests, new rapid tests have been developed to evaluate the potential of aggregates for AAR. The Oberholster and Davies hot 1N NaOH test appears to be a good, if severe, rapid test. A variation of this test has been used for the last three years in this laboratory, with generally good, but occasionally quirky results.

The AAR potential is measured by expansion. The test was performed on 19mm and 25mm diameter, 70mm length cores of mortar and concrete. The specimen size had little influence on test results. Consistent variability in expansion during the same test was seen in samples cored from the same block when all other conditions were constant. Additionally, variability in the expansion of the samples made from the same aggregate, the same mixture and the same curing condition but at different times was also evident. Small changes in procedure and/or conditions of the mixture resulted in significant changes in expansion. Different AR aggregate exhibited different rates of expansion, some were early rapid, some late rapid expanders. Thus expansion after a given length of exposure (14 days), or rate of expansion may not accurately evaluate the aggregate.

Contrary to expectations, pessimum effect was observed in the hot NaOH environment. An osmotic theory of AR expansion is offered as an explanation for the pessimum phenomenon.

Given all the variables that may affect the test outcome, it is suggested that every rapid test include two standards: non-reactive and reactive aggregate standard. All AAR expansions should be expressed both in terms of absolute and relative expansion to the two standards. The expansion readings should be adjusted to the mean expansion of the standard. Initial and final rates of expansion should also be used in evaluation.

2. INTRODUCTION

Alkali silica(te) reactivity (ASR) has been recognized as a growing problem in many areas of the world. Since ASR was first recognized in early 1940's [1], more aggregate types have been found to be susceptible. Also, as demand for aggregates rises, new, untested sources are being developed. AR has to be suspected in all materials that have silica or silicate content - even some carbonates with silicate minerals are suspect as alkali-silica reactive.

Alkali silica reaction is slow, but alkali silicate reaction even slower. The 'standard' ASTM and CSA tests that simulate the optimum natural conditions for alkali reactivity are also slow. However, when a new aggregate source is developed, the need for the material is immediate. Six to twelve month wait for test results is not acceptable. In some cases, concrete already placed may be questioned as to its reactivity. Taking large samples is expensive, and may not be possible without affecting the integrity of the structure.

A variety of rapid tests have been under development by different researchers during the last several years. Of these, the test proposed by Oberholster and Davies [2] appears to have a good correlation to the 'standard' tests. The rapid AR test described in the paper is a modification of the Oberholster and Davies test. The results obtained outline some advantages and some drawbacks with this test, and with similar accelerated tests in general.

2.1 Details of Testing Method:

The Oberholster test uses mortar bars of the ASTM C227 specification, and 1N NaOH immersion at 80° C to accelerate the alkali reactivity. This procedure was modified for use in this laboratory. The main modification involved the specimen size and the method of measuring, and is more fully described in [3]. Briefly, rather than casting three mortar bars for each test, a single block of either concrete or mortar is cast. After 24-h set, the block is de-moulded and cured for 24h in 80° C water. Three cores of either 19mm or 25mm diameter are cut from the block; the remainder of the block is stored moist for additional testing, if required. The cores are kept moist at all times, and are squared and 'dimpled' at ends to allow them to fit into the measuring equipment.

The equipment (Figure 1) consists of a double LVDT (linear variable differential transformer). One LVDT is used to set the original length of the sample, and the second reads the changes in length during the experiment. The sample length is measured seven times, and the 'cleaned' mean (to +/-1 standard deviation) is extracted. The means for the three cores per sample are likewise 'cleaned'. The final result is considered to be a very accurate measure of the sample's length. The accuracy is necessitated by the small length of the sample -about 70mm.

The cores are placed in the hot 1N NaOH environment for 2 days, cooled to room temperature to facilitate handling, and their length is measured. The cores are then returned to the hot environment; the cycle is repeated for a minimum of 14, and maximum of 21 days. The number or cycles depends on the nature of expansion observed.

2.2 Reactive Aggregates Tested:

Four types of reactive aggregates were used in this study. Three of these were obtained from the stockpile of reactive aggregates maintained by Ministry of Transportation, Ontario. They were alkali-silica carbonate from Spratt quarry near Ottawa, Ont., alkali-silicate crushed gravel from near Sudbury, Ontario, and alkali-carbonate reactive crushed stone from Kingston, Ontario. The fourth, an alkali-silica reactive aggregate was obtained from crushed chert cobbles from Putnam, Ontario (east of London). A non-reactive crushed limestone from research stockpiles at the University of Windsor was used as a control.

3. EXPERIMENTAL RESULTS

The experimental results presented here have been selected from along-term testing project of concrete and mortar containing reactive aggregate. Much of the testing involved treating aggregate, concrete, or mortar with various chemicals to test their effectiveness in controlling AR. In each of the tests an untreated control set is run; the results given here are collected from the control runs.

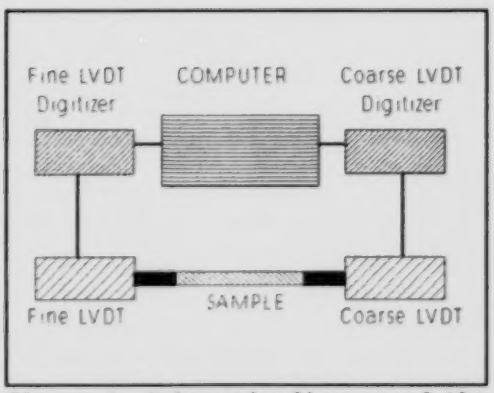

figure 1 Schematic diagram of the LVDT length measuring apparatus.

3.1 Comparison of Concrete and Mortar Expansion

The typical expansions obtained for concrete specimens containing -9.5mm graded aggregate are given in Figure 2. Similar results are shown in Figure 3 for mortar specimens

containing the same aggregate. The figures show a systematic parallelism of the curves which can be ascribed more to instrument drift than to actual changes in the expansion behaviour.

When the expansion of the concrete and mortar are compared (Figure 4), it is seen that at 18 day exposure, the mortar expands more in reactive alkali-silica(te) specimens than in does in reactive alkali-carbonate aggregate containing specimens. This type of behaviour was consistently noted in all experiments. Thus, although both concrete and mortar can be used to test for AR, somewhat different results may be expected, depending on the type of aggregate used. This is probably more of a non-equilibrium or rate of reaction problem dictated by the difference in the surface area of the reactive aggregate. The alkali-carbonate reaction involves the entire aggregate, whereas the alkali-silica reaction is more of a surface phenomenon. These results are only preliminary, and need to be investigated further.

3.2 Effect of specimen diameter on the rate of expansion.

The size (diameter) of specimen did not seem to have much effect on AR expansion. The results on two sizes, 19mm and 25mm diameter cores, are shown graphically in Figure 5. The work was done on concrete specimens cored from the same cast block, and shows expansion after 14 days of exposure. It is expected that similar outcome will be obtained on mortar specimens. This work suggests that smaller cores can be obtained from existing structures for rapid testing for AR, and the results will be comparable. Again, the data is preliminary, and more work needs to be done on a greater variety of sizes.

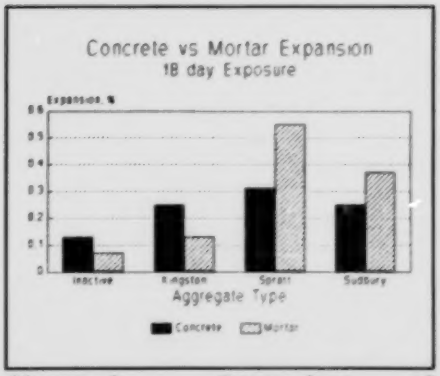

figure 4 Comparison of concrete and mortar expansions.

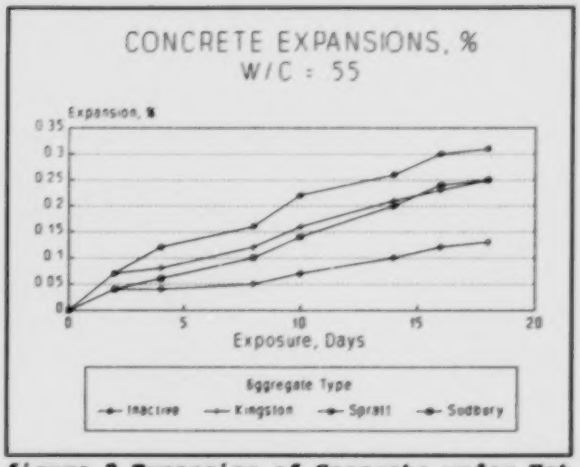

figure 2 Expansion of Concrete under Hot NaOH conditions.

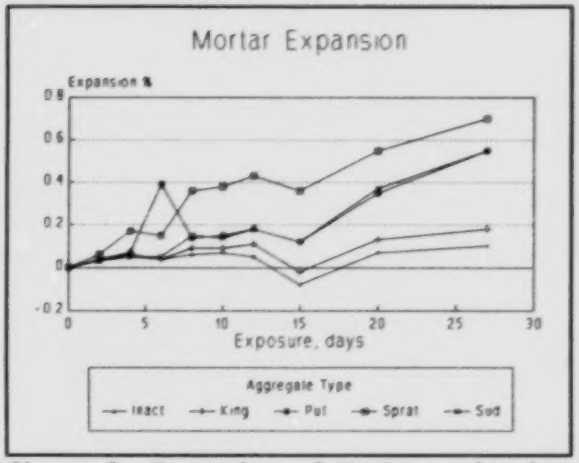

figure 3 Expansion of Mortar under hot NaOH conditions of exposure.

3.3 The Pessimum Effect.

The current understanding of the Pessimum Effect in AR holds that the AR expansion is controlled by both the amount of alkalies and the amount of reactive aggregate in the concrete - the alkali/reactive silica ratio [4]. For a given amount of alkali, if the amount of reactive aggregate is increased beyond certain level, the expansion falls off. If the amount of

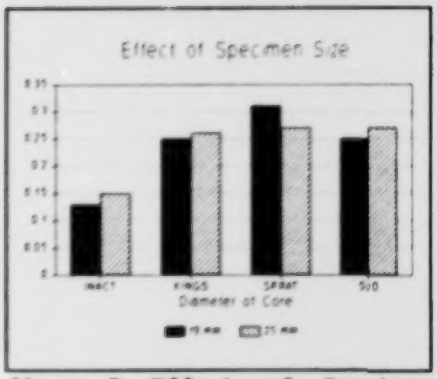

figure 5 Effect of Specimen size on the Rate of Reaction.

alkali is increased, the expansion will increase as the amount of reactive aggregate increases.

Thus, in rapid AR testing with abundant alkali in the form of hot NaOH solution, the convention would dictate that as the amount of reactive aggregate is increased, the expansion will also increase. This proved not to be the case, as is shown in Fig. 6.

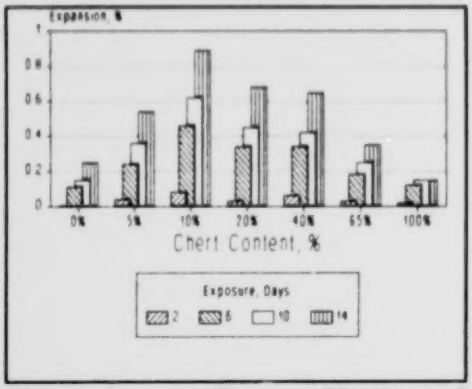

figure 6 The effect of increasing reactive chert content in mortar specimen.

The reasons for the pessimum effect need to be reconsidered. There is an overabundance of alkali in the accelerated hot NaOH environment, so the ratio of alkali/silica does not change significantly as the proportion of reactive silica is increased. According to currently held ideas, the expansion should continue, but perhaps at somewhat lower rate as the reactive silica proportion is increased. However, as Figure 6 shows, it does not. The explanation probably lies in the causes of expansion due to AR.

The AR expansion is produced by the alkali-rich gel imbibing low alkali water by osmosis. The amount of gel produced is indeed a function of the available reactive silica and the alkalies. However, the distribution and distance between the reactive silica particles within the mortar or concrete for a given content of alkalies is important. Several investigators have shown that by maintaining the alkali/reactive silica ratio constant while reducing the particle size, the expansion of the specimen increases [5,6]. Reducing the particle size increases the number of particles of reactive material in the mix, and decreases the distance between them.

If each particle produces silica gel, and if sufficient alkali is present to make that silica gel imbibe water, the particle can be considered as an expansive centre, deriving its water from the surrounding paste. Each particle can be considered to have an osmotic sphere of influence, as shown in the cartoon of Figure 7. As long as a high osmotic potential exists, particle will continue to produce expansive stress. If, however, there are sufficient number of particles present so that their spheres of influence overlap or interfere with each other, the osmotic differential will be decreased, and the water flow will be reduced or cease, as illustrated in the cartoon of Figure 8.

The above theory also explains the beneficial action of pozzolans in controlling expansion due to AR. The fine particles of the pozzolanic material can be thought of as individual centres around which an osmotic potential zone is set up. If the particles are in sufficient concentration so that the osmotic zones overlap, the osmotic difference within the concrete diminishes, and the expansion is prevented.

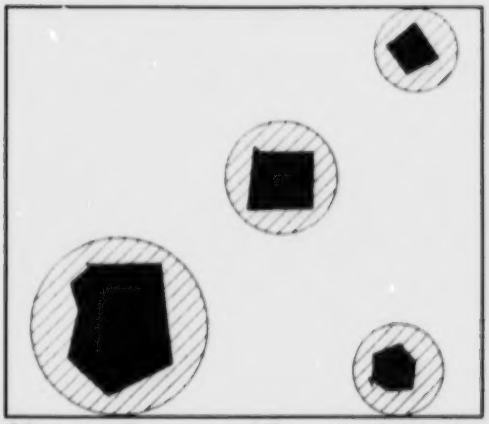

figure 7 AR aggregate concentration at pessimum or less: water is free to migrate to high potential osmotic zones surrounding each particle.

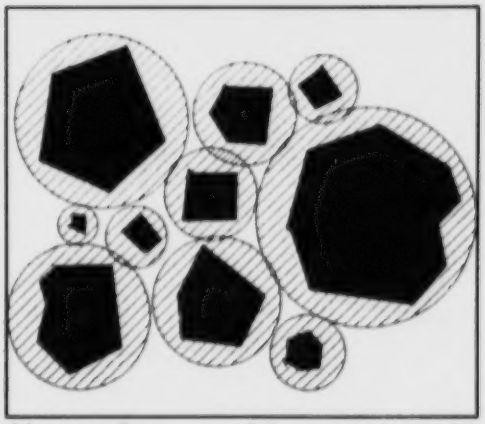

figure 8 AR aggregate concentration at greater than pessimum: overlapping high osmotic potential zones reduce osmotic gradient around each particle.

3.4 The reproducibility of the accelerated testing method.

For any testing method to be reliable, it must be reproducible -i.e., repeated tests on the same specimen must return comparable results. The rapid AR method described herein illustrates that, without comparison to standards, the reproducibility is not always achieved. Figure 9 gives the expansion of a non-reactive aggregate as measured in five different runs over a period of one year. Although the expansion is generally below that of an AR aggregate, there is, nevertheless, quite a variation in the results. Figures 10 and 11 below give the results on Kingston (ACR) and Sudbury (ASR) aggregate. In each case, there is considerable variation in the raw results. The small part of this variation is probably due to instrument drift as a result of temperature changes in the lab. However, the major departures from the norm are due to variations in the mixture proportions, curing conditions, and the conditions of testing.

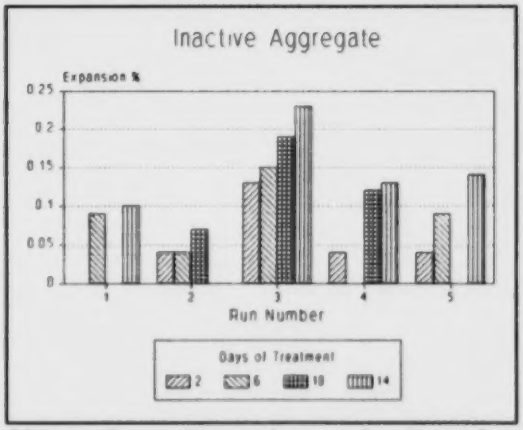

figure 9 Expansion of Inactive aggregate as measured over 5 runs.

Water-cement ratio, in so far as it controls the void ratio in the concrete and mortar, was found to significantly affect the expansion rate. W/C ratios of more than 0.55 decreased the AR expansion. The variations in conditions of testing were also found to be important. Not all of these have been identified. In one case, all AR and non-reactive specimens contracted throughout the test.

In this rapid AR test, the specimens are allowed to cool down to room temperature for measurement purposes. This appears to introduce an unknown variable to the procedure. In the next set of tests, the measurements in hot and cold state will be compared.

It has also been suggested by various workers that rather than the absolute expansion,

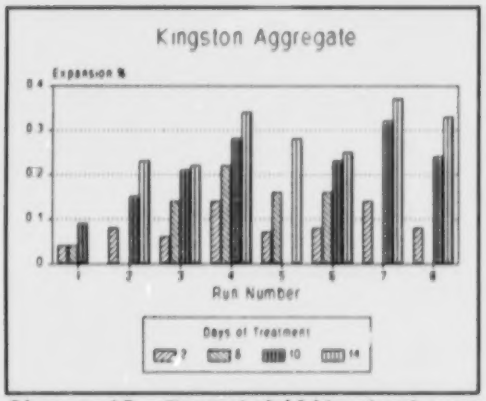

figure 10 Repeatability tests on Kingston (ACR) aggregate.

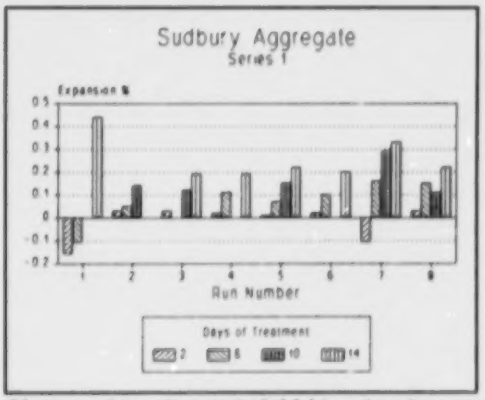

figure 11 Repeatability tests on Sudbury (ASR) aggregate.

the rate of expansion be used as a measure of AR. Figure 12 shows that there is a significant variation in the rate of expansion for different runs. The rate of expansion is very much aggregate-dependent. Some aggregates are slow early expanders (Sudbury), others are rapid early and slow later expanders (Putnam).

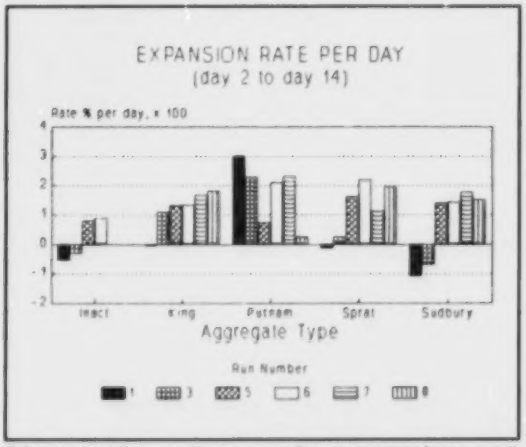

figure 12 Rate of expansion as measure of AR susceptibility.

The variation in the results obtained above would suggest that the rapid method as described here is not reliable. However, by adjusting the 'raw' data to that obtained from the 'inactive' standard, the results come to acceptably comparable levels. This emphasizes the need to run standards with each test. It is suggested that both inactive, and known AR active samples be measured with the unknown samples being tested. In this way, the variations in expansion due to specimen preparation, conditions of the test, and the testing method can be equalized.

The method of curing the specimen also has some influence on the results. Figure 13 shows the difference in expansion between the 'standard' 28-day curing of specimens containing ASR Putnam aggregate and the same mixture cured at 80° C and 40° C in water. As can be seen, the 28-day cure shows marginally greater expansion than does the rapid, one day cure methods at elevated temperatures. Rapid curing is therefore an acceptable way of speeding up the rapid test. Curing at 80° C has the added advantage of using the same oven settings for both curing and expansion testing.

4. CONCLUSIONS

The results presented above would suggest that rapid testing for Alkali Reactivity is viable, providing that considerable care is taken in minimizing variability of both sample preparation and

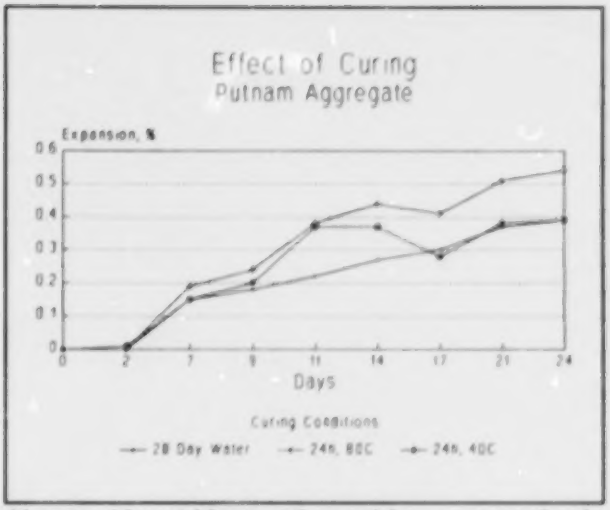

figure 13 Effect of rapid vs 'standard' curing methods on AR expansion in rapid tests.

conditions of testing. The following summarizes the findings:

1. Both concrete and mortar specimens can be used. Mortar gives somewhat greater expansion than does concrete.

2. Specimen size is not a factor in the rapid AR testing.

3. Void content of the specimen is one of the main controls of expansivity. Thus, the water/cement ratio, the physical process of mixing of the wet mortar or concrete, rodding or vibrating during casting (in so far as these affect the void ratio) must be strictly controlled.

4. Pessimum effect, although not expected in rapid NaOH-based AR testing, was found to be present. It is suggested that the distribution of the reactive particles in the mixture and the inter-particle distance between them rather than the alkali/reactive silica content govern the amount of expansion.

5. Aggregates of know response to AR testing should be used as part of each testing run, and the unknown aggregate material compared to them. Both inactive and active aggregates are suggested. Relative expansion to the standards rather than absolute numerical limits should be used to judge the unknown material.

6. The rapid AR testing method suggested here allows the use of relatively small specimens in the form of cores, cut from either the cast test block or from existing concrete structure. The remainder of the block can be used for either repeat tests or for other testing procedures.

5. REFERENCES

1. Stanton, T.E., The Expansion of Concrete Through Reaction Between Cement and Aggregate, Proc. Am. Soc. Civ. Engrs, 66, 1781-1811, 1940.

2. Oberholster, R. E., and Davies, G., An Accelerated Method for Testing the Potential Alkali Reactivity of Siliceous Aggregates, Cement and Concrete Research, 16, 181-189, 1986.

3. Hudec, P.P., and Larbi, J.A., A study of Alkali-Aggregate Reaction in Concrete: Measurement and Prevention, Part I: Measurement - Development and Prevention, Cement and Concrete Research, 19, 905-912, 1989.

4. Hobbs, D.W., Alkali Silica Reaction in Concrete, Thomas Telford, 22-25, 1988.

5. Hobbs, D.W. and Gutteridge, W.A., Particle Size of Aggregate and its Influence upon Expansion Caused by the Alkali-Silica Reaction. Mag. Concr. Res., 31, 235-242, 1979.

6. Diamond, S. and Thaulow, N., A Study of Expansion Due to Alkali-Silica Reaction as Conditions by the Grain Size of the Reactive Aggregate. Cement and Concrete Research,, 4, 591-607, 1974.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

EVALUATION OF A MODIFIED CHEMICAL METHOD TO DETERMINE THE ALKALI-REACTIVITY POTENTIAL OF SILICEOUS CARBONATE AGGREGATES

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ABSTRACT

This paper summarizes the results of laboratory work realized on a modified Chemical Method performed on the insoluble residues of siliceous limestone or dolostone aggregates to determine their alkali-reactivity potential in concrete. The influence of different parameters such as the concentration of the acid used for carbonate dissolution, and the particle size of the insoluble residues under test was first investigated. The precision of the adopted test procedure was then evaluated through an interlaboratory testing program involving 6 laboratories. Finally, the method was applied to more than 70 carbonate agggregates from the St.Lawrence Lowlands of Quebec and eastern Ontario.

1. INTRODUCTION

In the St.Lawrence Lowlands (Quebec, Canada), limestones, dolostones and sandstones of Ordovician Age are the main source of concrete aggregates. Some siliceous limestones exploited in the Ottawa, Montreal, Trois-Rivieres, and Quebec City areas have already been recognized as alkali-silica reactive in concrete [1-9]. The standard ASTM C289 Chemical Method [10] proved to be unreliable for the determination of the potential alkali-reactivity of these carbonate aggregates since they are all classified as non deleterious [3,6,11]. This behavior is mainly attributed to the presence of calcite which surrounds and thus protects the reactive insoluble residues from the alkaline attack, while also inducing errors in the measurements of Sc and Rc values. Indeed, Mielenz & Benton [12] reported that: *"calcium released by calcite combine with the dissolved silica causes precipitation of calcium silicate with absorbed or combined alkalies"*, sodium in the case of the Quick Chemical Test [13]. This behavior is confirmed by the precipitation of white gels containing silica, calcium and sodium at the surface of rock cylinders cored from potentially reactive siliceous limestones from the Quebec City area [6]. In order to prevent carbonate interference, a modified version of the ASTM C289 Chemical Method was developed [3]. The test is done on the insoluble residues of the limestone or dolostone aggregates, after dissolution of the carbonates with concentrated hydrochloric acid. This modified chemical method (on insoluble residues) is widely used in the Quebec Province and has been recently proposed by the CSA Sub-Committee A5 as a standard

procedure for the chemical determination of the potential alkali-silica reactivity of quarried carbonate aggregates exploited in the St.Lawrence Lowlands of Quebec and Ontario. This paper summarizes the results of laboratory work performed to investigate the reliability of this testing method for the purpose mentioned above.

2. MATERIALS AND METHODS

2.1 Geographical Location and Geological Association of the Investigated Quarries and Samples

The St. Lawrence Lowlands mainly consist of relatively unfolded and flat-lying beds of limestones, dolostones, sandstones and shales, extending on both sides of the St. Lawrence River. These are bordered to the northwest by the Precambrian Shield, along a series of normal faults, and to the southeast by the folded rocks of the Appalachian belt [14]. 71 samples were collected (bed-by-bed sampling) from twenty-two quarries selected to represent the lateral and vertical variations within this large structural domain. Lithostratigraphic correlations were mainly based on petrographic considerations, but also on stratigraphic markers consisting of beds of altered volcanic ashes called "K-Bentonites"[15]. Figure 1 and Table 1 present the location of the quarries, the geological association, as well as the petrographic characteristics of the samples.

2.2 Experimental

Figure 2 illustrates the detailed experimental procedure followed in this research program. For each of the 71 samples selected (Figure 1), and in accordance with ASTM C289, about 2 kg of 150-300 micrometers particles were produced by progressively crushing a representative 5 kg subsample of coarse aggregate particles (5-20 mm). The influence on the Rc and Sc values measured at the Chemical Test of parameters such as the acid concentration used for carbonate dissolution, and the insoluble particle size distribution was first determined (***PART 1***). A subgroup of 14 aggregates representative of various petrographic facies were selected for this part of the project, for which insoluble residues were obtained after complete carbonate dissolution in concentrated HCl (12N) and diluted HCl (1.2N) of 150-300 um representative subsamples (Figure 2). The suspension was either washed with tap water over a no. 50 sieve to retain only the 150-300 um insoluble material, or filtered and washed with distilled water to obtain the total insoluble residue (0-300 um). In all cases, the oven dried residue was then tested in accordance with ASTM C289, except that 12.5 g of material was used (2 subsamples) instead of 25 g (3 subsamples), because of the high porosity of the insoluble residue. A final test procedure was then adopted, and its precision evaluated through an interlaboratory test program (***PART II***). In this case, 5 kg samples (5-20 mm) of 6 carbonate aggregates, 1 sandy and dolomitic limestone (M8), 1 siliceous dolostone (K1), 3 siliceous limestones (C5, V1 & W1), and 1 non reactive reference limestone (Z1), were sent to the 6 participating laboratories. Parameters like the crushing efficiency, the percentage of insoluble residues, and the values of dissolved silica (Sc) were investigated. Finally, the adopted test procedure was applied to the remaining samples previously collected in the St.Lawrence Lowlands (***PART III***).

On a reference basis, concrete prisms were made (75 mm x 75 mm x 300 mm) using a type 10 high alkali cement (1.0 Na_2O equiv.), and an innocuous granitic sand. The aggregate:sand:cement:water mix proportions were of 3.18 : 2.19 : 1 : 0.50, with a cement content of 350 kg/m^3. The total alkali content of the mix was increased to 1.25% (Na_2O equiv.) by the addition of NaOH. The concrete prisms were stored at 38°C and 100% R.H., with length change measurements made at specified times using a vertical comparator readable to 0.001mm (CSA

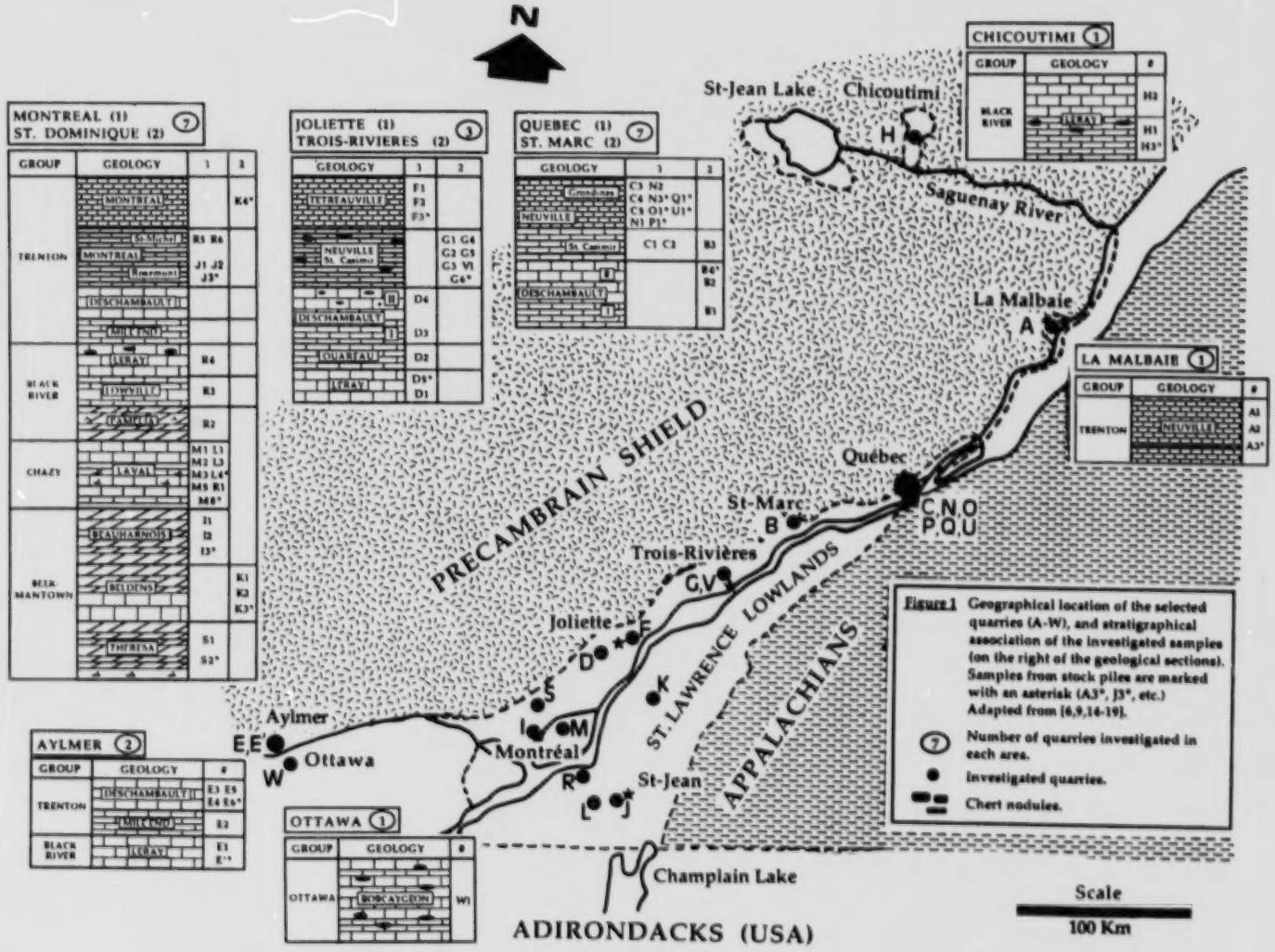

N
MONTREAL (1)
ST. DOMINIQUE (2)
7
GROUP
GEOLOGY
TRENTON
MONTREAL
St-Michel
Bourmont
DESCHAMBAULT II
MELBOURNE
BLACK RIVER
LERAY
LOWVILLE
ROCKLAND
CHAZY
LAVAL
BEKMANTOWN
BEAUHARNOIS
BELDENS
THERESA
JOLIETTE (1)
TROIS-RIVIERES (2)
3
TETREAUVILLE
NEUVILLE
St. Casimir
DESCHAMBAULT
COURTEAU
QUEBEC (1)
ST. MARC (2)
7
Grondines
NEUVILLE
St. Casimir
DESCHAMBAULT
CHICOUTIMI (1)
BLACK RIVER
LERAY
St-Jean Lake
Chicoutimi
Saguenay River
La Malbaie
A
LA MALBAIE (1)
TRENTON
NEUVILLE
Québec
St-Marc
B
C,N,O
P,Q,U
Trois-Rivières
G,V
Joliette
D
E
S
F
St. Lawrence Lowlands
Appalachians
Aylmer
E,E'
W
Ottawa
Montréal
I
M
R
St-Jean
L
J
AYLMER (2)
TRENTON
DESCHAMBAULT II
MELBOURNE
BLACK RIVER
LERAY
OTTAWA (1)
OTTAWA
BOBCAYGEON
Champlain Lake
ADIRONDACKS (USA)
Scale
100 Km
Figure 1 Geographical location of the selected quarries (A-W), and stratigraphical association of the investigated samples (on the right of the geological sections). Samples from stock piles are marked with an asterisk (A3*, J3*, etc.) Adapted from [6,9,14-19].
Number of quarries investigated in each area.
Investigated quarries.
Chert nodules.

TABLE 1: Petrography of the Geological Formations or Members Investigated.

Formation (Member)	Petrography
Tétreauville Neuville (Grondines)	Fine grained, micritic and dark argillaceous limestone, thin bedded (5 - 15 cm), with numerous shale interbeds (sometimes more than 25% of the global sequence).
Neuville (St. Casimir)	Fine to medium grained and medium grey siliceous limestone, medium bedded (15 - 30 cm); occasional nodules of dark grey chert.
Montréal (Rosemont) (St. Michel)	Fine to medium grained, dark grey, siliceous and argillaceous limestone, thin bedded; also dark grey and medium grained skeletal limestone, medium bedded.
Deschambault	Fine grained, medium bedded and medium grey siliceous limestone (I); Coarse grained, pale grey to beige, high-calcium and thick bedded (> 30 cm) skeletal limestone (II); occasional nodules of black chert.
Mile End Ouareau	Fine to medium grained, medium grey, micritic and shelly limestone, medium to thick bedded.
Leray Bobcaygeon	Fine grained and medium grey, sometimes brownish, medium bedded and micritic limestone; occ. nodules of chert.
Lowville	Fine grained and massive bluish-grey limestone, medium bedded; "bird eyes" texture; high-calcium content.
Pamélia	Fine to medium grained and massive dolostone, greenish-grey, medium to thick bedded, locally calcareous and sandy; characteristic "orange-weathering" surface color.
Laval (St. Martin)	Coarse grained and pale-grey skeletal limestone, more or less dolomitic, sandy or oolithic, thick-bedded; also, dark grey, fine grained sandstone, massive bedding difficult to separate; some levels contain large amounts of shales.
Beauharnois	Dense to crystalline dolostone, sometimes laminated, fine to medium grained, various colors, medium to thick bedded; pockets filled with white calcite and gypsum, pink dolomite.
Beldens	Massive, fine grained, thick bedded, high-calcium and pale grey limestone; also, dark grey and fine grained dolostone, thick bedded, "orange weathering" surface color.
Theresa	Fine to medium grained, medium grey dolostone, thick bedded; interlayed fine grained and dark grey sandstone, in thin layers of a few centimeters.

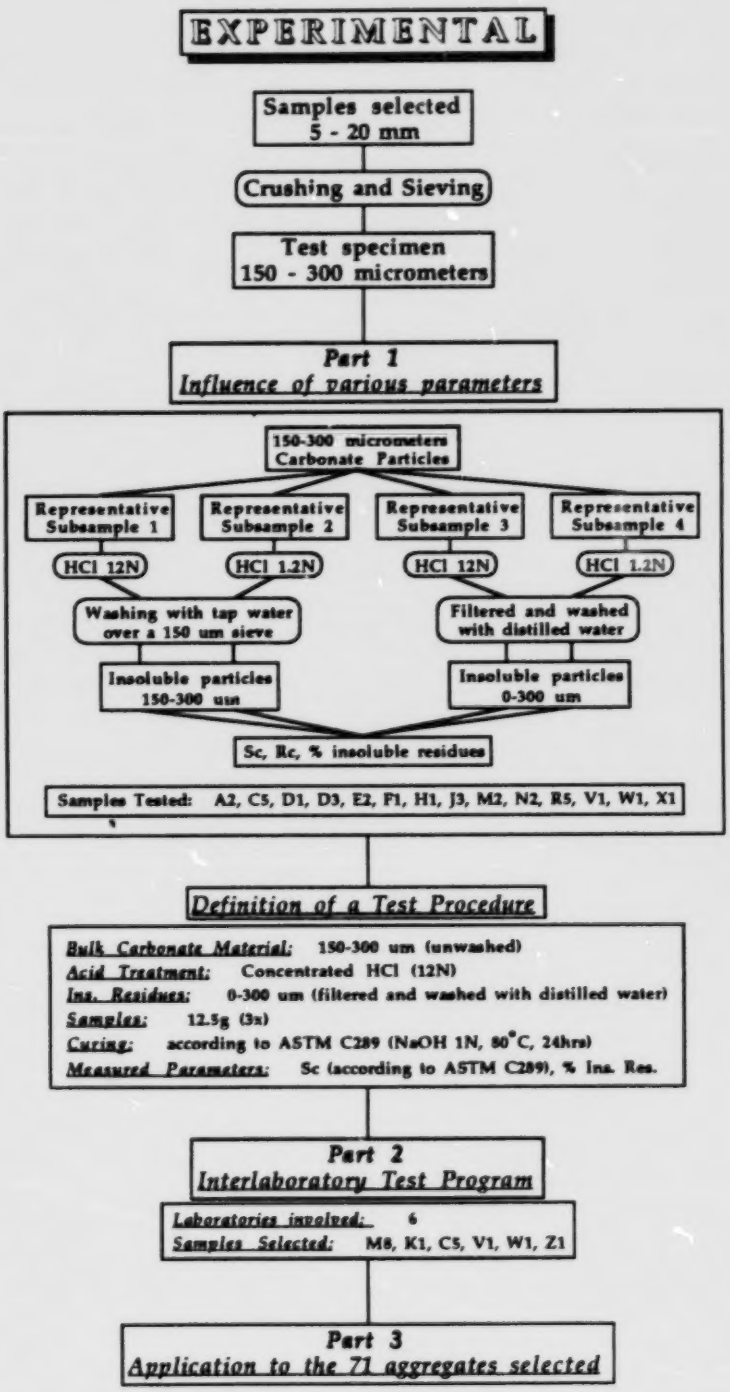

EXPERIMENTAL
Samples selected
5 - 20 mm
Crushing and Sieving
Test specimen
150 - 300 micrometers
Part 1
Influence of various parameters
150-300 micrometers
Carbonate Particles
Representative Subsample 1
Representative Subsample 2
Representative Subsample 3
Representative Subsample 4
HCl 12N
HCl 1.2N
HCl 12N
HCl 1.2N
Washing with tap water over a 150 um sieve
Filtered and washed with distilled water
Insoluble particles 150-300 um
Insoluble particles 0-300 um
Sc, Rc, % insoluble residues
Samples Tested: A2, C5, D1, D3, E2, F1, H1, J3, M2, N2, R5, V1, W1, X1
Definition of a Test Procedure
Bulk Carbonate Material: 150-300 um (unwashed)
Acid Treatment: Concentrated HCl (12N)
Ins. Residues: 0-300 um (filtered and washed with distilled water)
Samples: 12.5g (3x)
Curing: according to ASTM C289 (NaOH 1N, 80°C, 24hrs)
Measured Parameters: Sc (according to ASTM C289), % Ins. Res.
Part 2
Interlaboratory Test Program
Laboratories involved: 6
Samples Selected: M8, K1, C5, V1, W1, Z1
Part 3
Application to the 71 aggregates selected

Figure 2

Standards). An expansion limit of 0.04% at 6 months is chosen to distinguish reactive or potentially reactive aggregates from the non reactive ones. This value was obtained by comparing test results obtained on another series of concrete prisms tested in accordance with the CSA standards (cement content of 310 kg/m^3). It is noted that the 0.04% of expansion generally corresponds to the detection of visible cracks at the surface of the concrete prisms.

3. RESULTS AND DISCUSSION

3.1 Influence of Various Parameters on the Test Results - *PART I*

The Sc, Rc and Sc/Rc values obtained at the Modified Chemical Test (on insoluble residues) for each of the four subsamples tested (HCl 12N, 150-300 um; HCl 12N, 0-300 um; HCl 1.2N, 150-300 um; HCl 1.2N, 0-300 um; Figure 2) are presented on Table 2, together with the corresponding 6 month expansion values obtained in the Concrete Prism Test. The amounts of insoluble residues obtained for each of these subsamples is also indicated on Table 2 (in italic). For example, for the aggregate E2, about 600 g of 150-300 um carbonate particles (subsample #1) have been placed in concentrated HCl (12N) until complete carbonate dissolution. The supension was then washed over a 150 um sieve with tap water and then put in an oven (110^{o}C. About 33 g of 150-300 um insoluble particles were thus recovered, corresponding to 5.5% of the starting material.

3.1.1 Effect of washing the aggregate particles (150-300um) before the acid attack

For three selected aggregates showing various expansivity level in the Concrete Prism Test (C5, V1 and E2), a sub-sample of the bulk material used in the chemical test (300-150 um aggregate particles) was washed with tap water over a 150 um sieve prior to the acid treatment with concentrated HCl ("washed" on Table 2). No significant difference for the Sc and Rc values subsequently obtained in the Chemical Test was observed compare to the unwashed samples, thus indicating that it is not necessary to wash the limestone particles before the acid treatment. However, it is recommended to add just enough distilled water to moisten the limestone particles prior to the treatment with concentrated HCl. This minimizes excessive effervescence, and also reduces the induced-extraction of water molecules from the crystalline structure of certain clay minerals (A. Chagnon, INRS Georessources, personal communication).

3.1.2 Effect of the concentration of the acid used for carbonate dissolution, and of the insoluble particle size distribution.

Despite no strong trend for the Sc, Rc or Sc/Rc values measured in the Modified Chemical Test in Table 2, some general tendencies can be noted. Indeed, for each acid concentration, as illustrated by the vertical lines on figure 3a, samples of total insoluble residues (0-300 um) generally give higher Rc values than those composed of 150-300 particles. However, for a given insoluble size fraction (as illustrated by the vertical lines on figure 3b), there is no evidence that concentrated HCl produces higher Rc values than diluted HCl. Concerning the Sc values, for each particle size fraction, there is a slight tendency to higher Sc values when concentrated HCl is used for carbonate dissolution (illustrated by the vertical lines on figure 4a). However, for each acid concentration (as illustrated by the vertical lines on figure 4b), total insoluble residues (0-300 um) tend to produce higher Sc values than the 150-300 um residues with diluted HCl, while the reverse tendency is observed but less evidently with concentrated HCl. According to these observations, and considering that the carbonate dissolution is much faster in concentrated than in diluted HCl, it appears that the insoluble

TABLE 2: Experimental Results - PART I

Sample	HCl 12N						HCl 10%						INS. RES. %[1]	Concrete Prism Test Expansion %[2]
	-300+150 um			-300 um			-300+150 um			-300 um				
	Sc	Rc	Sc/Rc	Sc	Rc	Sc/Rc	Sc	Rc	Sc/Rc	Sc	Rc	Sc/Rc		
E2	107[3]	76[4]	1.41	88	107	1.14	97	85	0.82	77	114	0.68	10.7	0.011
	5.5[5]			*10.7*			*4.4*			*10.7*				
washed	116	91	1.27	103	113	0.91								
	5.5			*10.5*										
M2	29	34	0.85	42	46	0.27	16	59	0.91	27	45	0.60	19.8	0.021
	13.6			*19.7*			*13.7*			*19.4*				
H1	222	92	2.41	190	145	2.26	199	88	1.31	274	114	2.40	6.0	0.034
	2.5			*5.4*			*2.6*			*5.9*				
R5	119	112	1.06	120	139	1.26	97	77	0.86	82	151	0.54	14.0	0.043
	n.a			*n.a*			*7.4*			*13.9*				
F1	208	108	1.93	206	141	1.09	151	138	1.46	237	150	1.58	14.7	0.061
	8.3			*15.0*			*7.6*			*14.4*				
C5	544	117	4.65	523	194	2.15	357	166	2.70	472	180	2.62	11.8	0.074
	n.a			*11.5*			*n.a*			*n.a*				
washed	506	118	4.29	563	150	3.75								
	3.9			*11.5*										
J3	170	114	1.49	144	138	1.67	155	93	1.04	129	118	1.09	12.0	0.081
	5.7			*11.9*			*4.4*			*11.8*				
A2	524	88	5.95	593	116	18.5	500	27	5.11	541	126	4.29	14.3	0.108
	n.a			*13.5*			*7.8*			*13.8*				
D3	—	—	—	317	155	3.49	370	106	2.05	293	143	2.05	6.3	0.158
	n.a			*5.9*			*1.6*			*6.2*				
N2	491	145	3.36	440	177	2.77	405	146	2.49	397	184	2.16	15.8	0.159
	9.5			*17.4*			*8.5*			*17.3*				
D1	242	75	3.23	320	113	2.65	254	96	2.83	304	112	2.71	11.6	0.190
	6.1			*11.5*			*6.2*			*11.5*				
V1	252	104	2.42	380	158	1.51	187	124	2.41	224	151	1.48	11.5	0.268
	n.a			*n.a*			*n.a*			*n.a*				
washed	276	111	2.49	368	157	2.34								
	4.4			*11.1*										
W1	657	72	9.13	620	110	6.42	610	95	5.64	621	107	5.80	10.4	0.274
	4.5			*9.8*			*4.2*			*9.8*				
X1	101	47	2.15	116	44	2.64	106	36	2.94	109	46	2.37	88.9	n.a
	n.a			*n.a*			*82.7*			*87.7*				

1 Percent of insoluble residues determined on 5g of bulk material (unwashed 150-300 um particles) after complete carbonate dissolution with concentrated HCl.

2 Expansion values of concrete prisms (cement content: 350kg/m^3, alkali content raised to 1.25% Na_2O equivalent) after 6 months of curing at 38°C and 100% R.H.

3 Values of Dissolved Silica in millimoles per liter (average of 2 measurements).

4 Values of Reduction in Alkalinity in millimoles per liter (average of 2 measurements).

5 Amount of the insoluble size fraction considered in the sub-sample tested.

residues may be as much affected (in terms of Rc and Sc values) by a short stay in concentrated HCl than by a longer stay in diluted acid.

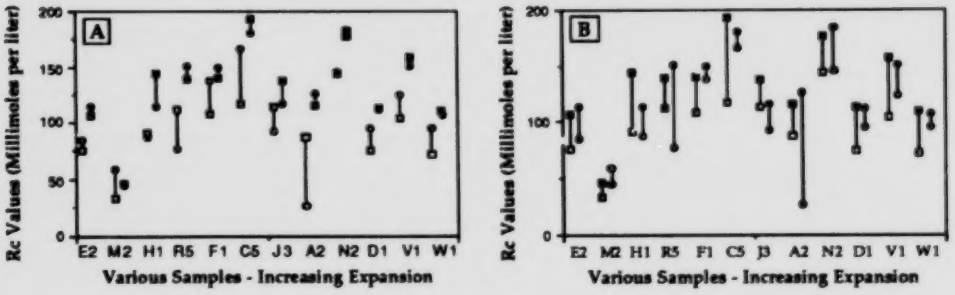


Figure 3: Effect of the acid concentration (HCl 12N vs 1.2N) used for carbonate dissolution (A), and of the insoluble particle size distribution (0-300 vs 150-300 um)(B) on the Rc values measured in the Modified Chemical Method. The results are classified according to the amount of expansion measured in the Concrete Prism Test.

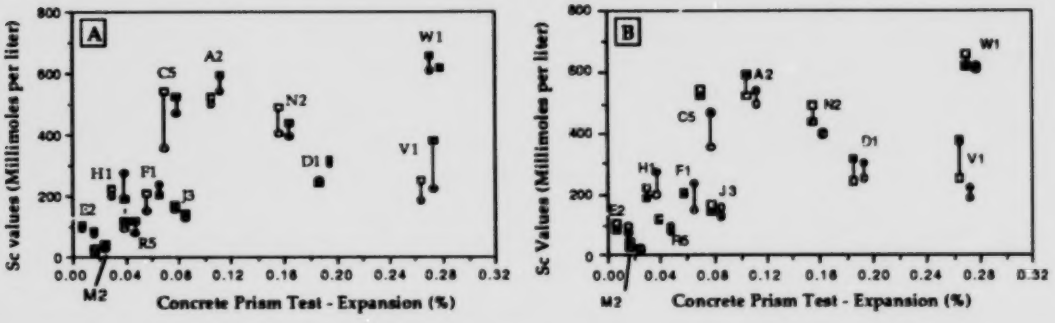


Figure 4: Effect of the acid concentration (HCl 12N vs 1.2N) used for carbonate dissolution (A), and of the insoluble particle size distribution (0-300 vs 150-300 um)(B) on the Sc values measured in the Modified Chemical Method. The results are classified according to the amount of expansion measured in the Concrete Prism Test.

From the amount of insoluble residues (0-300 um or 150-300 um) measured in each sub-sample tested (values in italic on Table 2), it can be calculated that the 150-300 um insoluble particles generally represent from 26 to 57% of the total insoluble residue (0-300 um). For a sandstone of the Lorraine Group from the Quebec City area (X1), and a sandy limestone (M2), this value increases significantly to 94% and 70%, respectively. On an average, the Sc values obtained for the two insoluble particle sizes tested are not significantly different (at a given acid concentration). This suggests that the specific area of the total insoluble residue (0-300 um) is not significantly higher than the 150-300 um insoluble material despite a large proportion of particles < 150 um in the former material (specific area measurements in progress to verify this hypothesis). Indeed, in the reactive siliceous limestones from the Ottawa, Trois-Rivières and Quebec City areas, crypto- to microcrystalline quartz forms a porous network surrounding microsparite grains averaging around 10 to 20 micrometers in size [3,4,6,11,20,21].

3.1.3 Relationship between the Modified Chemical Method and the Concrete Prism Test.

From the results presented on Table 2 and Figures 3 & 4, neither the Rc or the Sc values, nor the Sc/Rc ratio show a clear correlation with the amount of expansion in the Concrete Prism Test. This confirms one of the conclusions mentioned by Mielenz et al. [22]: "*the amount of silica dissolved is related only in general to deleteriousness, and reduction in alkalinity alone is no clue to deleteriousness*". When the chemical test results are plotted on the ASTM C289 standard graph (figures 5a-d), "grey-zones" can be drawn where expansive and non-expansive samples fall together. The best results are for total insoluble residues (0-300) obtained with concentrated HCl. In this case, samples with Sc values < 120 mm/l are non expansive in the Concrete Prism Test (< 0.04%), while those with Sc > 200 mm/l are expansive. A "grey-zone" is thus observed for Sc values between 120-200 mm/l. Vivian [23] mentioned that, when the amount of dissolved silica exceeds 100 millimoles per liter, the rock or aggregate involved should contain a sufficient amount of reactive material to produce significant expansion in concrete. Thaulow & Olafsson [24] found the 100 mm/l limit useful to establish the potential for reactivity of some Danish and Icelandic sands. However, Sims [25] reported that many aggregates exploited in the UK, especially the Thames Valley flint gravels, give Sc values exceeding 100 mm/l while being non deleterious in concrete structures. Grattan-Bellew [26] reported the following limits for Danish sands containing flint, and Icelandic sands with volcanic glass:

Reactive Aggregate	Non deleterious (mmoles/liter)	Pot. deleterious (mmoles/liter)	Deleterious (mmoles/liter)
Sand with flint	Sc < 50	50 < Sc < 200	Sc > 200
Sand with vol. glass	Sc < 100	100 < Sc < 200	Sc > 200

3.1.4 Adopted final test procedure

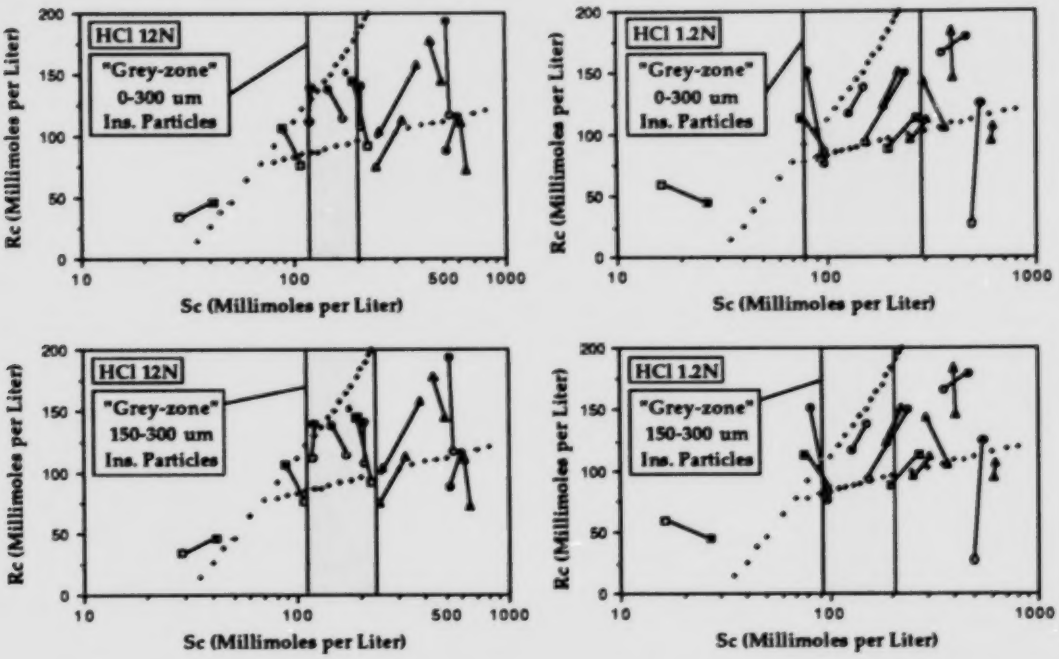
From the results presented above, a final test procedure involving the acid treatment of unwashed 150-300 um bulk limestone or dolostone particles with concentrated HCl (12N) was adopted. The total residues (0-300 um) recovered after adequate washing and filtering the "acid" suspension, and dried in an oven at 110°C, are then used to measure Sc values as described in the standard procedure ASTM C289 [10]. It was not recommended at that time of the project to measure the Rc values since that parameter did not seem to give useful additional informations. The percent of insoluble residue must be measured on each sample tested (see section 3.2.3).

3.2 Interlaboratory Study on the Modified Chemical Method- *PART II*

The precision of the adopted test procedure mentioned above was evaluated from the results obtained by 6 laboratories on 6 selected aggregates (Figure 2) [27].

3.2.1 Crushing efficiency and percentage of insoluble residues

The crushing and sieving operations required for the preparation of the ASTM C289 test specimen certainly represent a critical step in the test procedure. Indeed, the amount of 150-300 um particles must be optimized to obtain a subsample representative of the investigated coarse aggregate. Important variations in the crushing efficiency are observed between the various



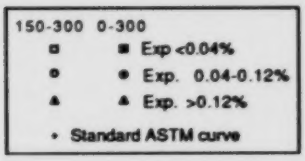


Figure 5: Effect of the acid concentration (HCl 12N vs 1.2N) used for carbonate dissolution, and of the insoluble particle size distribution (0-300 vs 150-300 um) on the Sc and Rc values measured in the Modified Chemical Method. The results are reported on the standard graph ASTM C289. "Grey- zones" of various width are observed where non reactive and reactive aggregates fall together.

participants in this study (Figure 6), but also between the aggregates tested. However, the procedure used in three laboratories (# 2, 3 & 4) seems consistent as suggested by the relatively parallel curves obtained on the various aggregates.

The percentages of insoluble residues measured by each laboratory on the various investigated aggregates are illustrated on Figure 7. Relatively high coefficient of variations are observed for aggregates V1 and Z1, while some individual results largely deviate from the average. More acurate results could be obtained by determining the % of insoluble residues on smaller subsamples. Such values should then be compared to the results obtained in the present test procedure to verify if the carbonate dissolution has been completed, or if some material may have been lost during the washing and filtering operations.

3.2.2 Dissolved Silica (Sc) values and Effect of the analytical method

The amounts of dissolved silica (Sc) measured after 24 hours of immersion in a 1N NaOH solution at 80°C are illustrated on Figure 8. It must be noted that different analytical methods (Photometry, Gravimetry and Atomic absorption) were used by the different laboratories for the determination of the Sc values. High coefficient of variations were observed, especially for the 3 aggregates showing low values of dissolved silica. Two of these

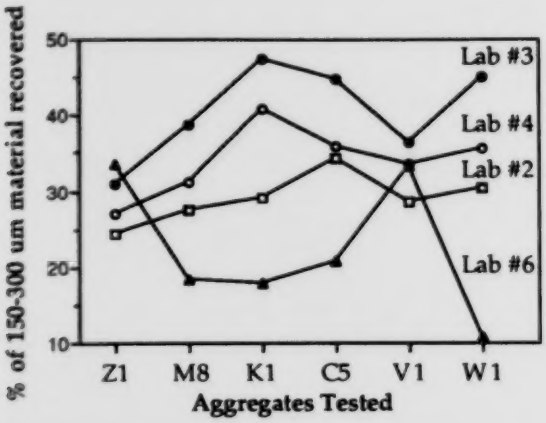
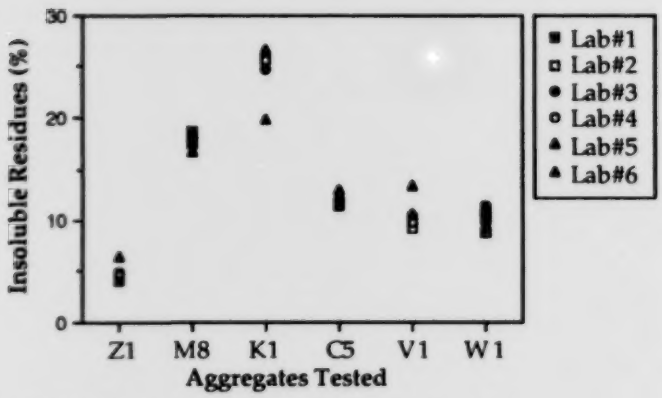


Figure 6: Variations in the crushing efficiency obtained for six aggregates retained for the Interlaboratory Test Program.

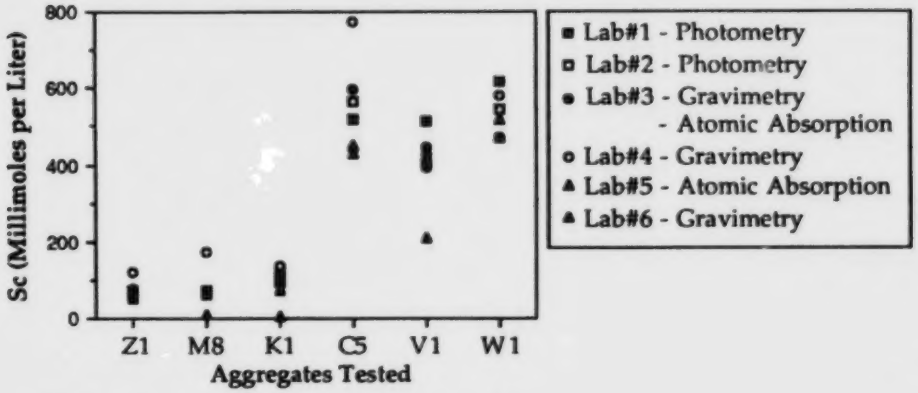


	Z1	M8	K1	C5	V1	W1
AVG.	4.8	17.8	24.7	11.8	10.4	10.0
STD.	0.9	0.7	2.5	0.6	1.6	1.0
VAR.	19%	4%	10%	5%	15%	10%

Figure 7: Variations in the percentage of insoluble residues measured on the 6 aggregates retained for the Interlaboratory Test Program. (AVG: Average; STD: Standard Deviation; VAR: Coefficient of Variation).

aggregates (M8 & K1) contain dolomite as a primary or secondary constituent. This mineral has already been recognized as susceptible to cause unreliable results in the Chemical Test [12]. The method itself used for chemical analysis cannot explain the large variations observed, since 1) the results corresponding to each particular method are randomly distributed with respect to the average values measured, 2) the results obtained on parallel measurements realized by gravimetric and atomic absorption methods (Lab #3) are very similar [27], and 3) previous tests realized at Laval University gave very similar Sc values when measured by atomic absorption and photometric methods [21]. It is noted that when we use a 200 millimoles per liter limit (as observed on figure 5a), all the laboratories classify the well-known reactive aggregates C5, V1 and W1 as effectively deleterious. On the other hand, non reactive aggregates M8 and K1 fall

in the "grey-zone" associated to potentially deleterious aggregates (interval 120-200 millimoles per liter on figure 5a) at least once.



	Z1	M8	K1	C5	V1	W1
AVG.	78	76	85	556	399	534
STD.	22	53	45	125	101	60
VAR.	29%	70%	53%	23%	25%	11%

Figure 8: Variations in the values of Dissolved Silica (Sc) measured in the Modified Chemical Test by the different laboratories involved in the Interlaboratory Test Program.
(AVG: Average; STD: Standard Deviation; VAR: Coefficient of Variation).

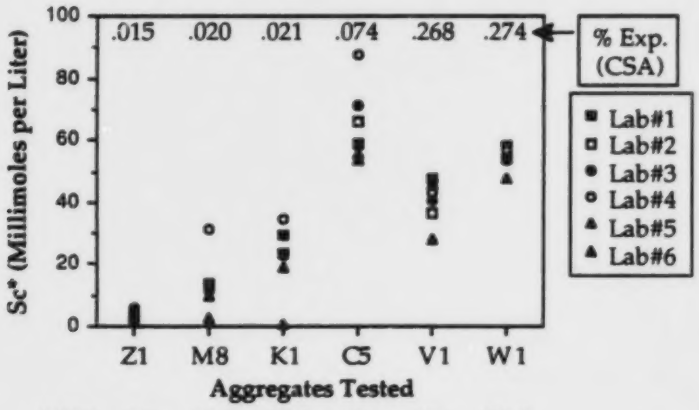
3.2.3 Corrected Dissolved Silica (Sc*)

Corrected Sc values (Sc*) are calculated by multiplying the bulk Sc values by the % of insoluble residues in the investigated samples. This parameter is proposed in order to compensate for the fact that the test is performed on 100% insoluble residues, and that this material forms only a minor proportion of the investigated aggregates. Since large differences are obtained by the different laboratories for the Sc values and the % of insoluble residues, the corrected Sc values evidently present similar variations (Figure 9). The Sc* values do correlate very well with the amount of expansion measured in the Concrete Prism Test for the investigated aggregates (Figure 9).

3.3 Application to the Carbonate Aggregates of the St.Lawrence Lowlands - *PART III*

Figures 10 and 11 present the Sc, Rc, Sc* and Rc* values obtained in the Modified Chemical Test for the 71 aggregates sampled all along the St.Lawrence Lowlands (Figure 1). The results are grouped in accordance with the geological association of these aggregates, and their behavior at the Concrete Prism expansion Test. Empty and filled symbols correspond to non expansive (exp. < 0.04% at 6 months) and expansive aggregates (exp. > 0.04%), respectively. Of the 71 aggregates tested, 35 were found expansive in the Concrete Prism Test (Table 3), all corresponding to siliceous limestones of the Trenton Group (74% - 32/43) and of the Black River Group (26% - 3/11). Indeed, none of the investigated aggregates belonging to the Chazy and Beekmantown Groups produced concrete expansions higher than the 0.04% limit, and are consequently considered non reactive (Table 3). In addition, among all the aggregates

tested, none with an amount of insoluble residues lower than 5% gave concrete expansions higher than the 0.04% limit.



AVG.	3.7	13.5	21.8	65.4	40.4	52.8
STD.	1.4	9.6	11.6	12.8	7.2	4.5
VAR	37%	71%	53%	20%	18%	8%

Figure 9: Variations in the values of Corrected Dissolved Silica (Sc*) calculated from the results obtained at the Modified Chemical Test by the different laboratories involved in the test program. (AVG: Average; STD: Standard Deviation; VAR: Coefficient of Variation).

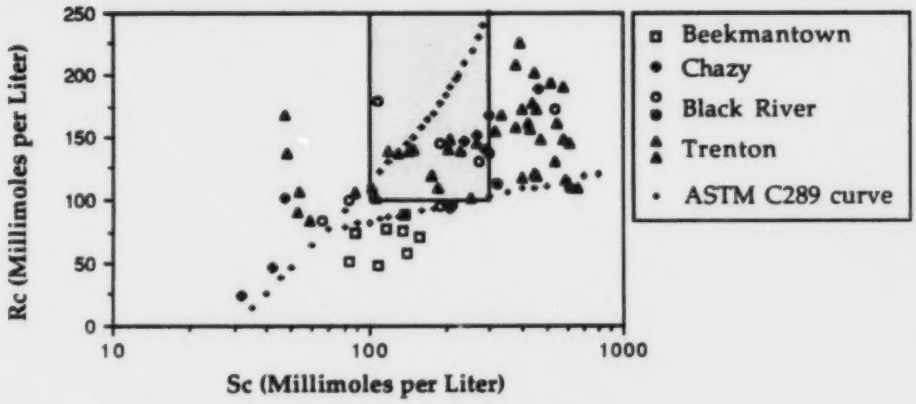


Figure 10: Values of Reduction in Alkalinity (Rc) and Dissolved Silica (Sc) measured at the Modified Chemical Test for the various aggregates sampled in the St. Lawrence Lowlands.The samples are grouped according to their geological association and their behavior at the Concrete Prism Test. (**Empty symbols**: non reactive aggregates - exp. < 0.04%; **Filled symbols**: Reactive aggregates - exp. > 0.04%).

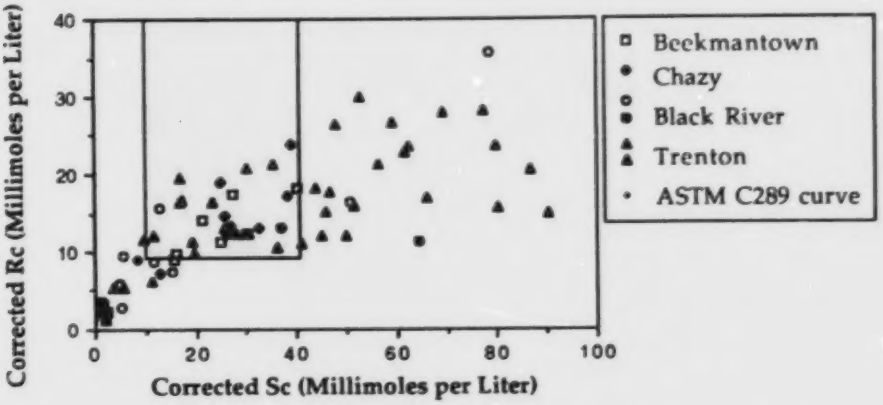


Figure 11: Corrected Rc and Sc values calculated from the results obtained in the Modified Chemical Test for the various aggregates collected in the St. Lawrence Lowlands.The samples are grouped according to their geological association and their behavior in the Concrete Prism Test. (**Empty symbols**: non reactive aggregates - exp. < 0.04%; **Filled symbols**: Reactive aggregates - exp. > 0.04%).

Some useful limits can be drawn from the results presented on Figures 10 & 11 and on Table 3. First, all the samples (30) meeting at least one of the following "limit values": Sc < 100 mm/l, Rc < 100 mm/l, Sc* < 10 mm/l, Rc* < 9.5 mm/l, are non expansive aggregates (Exp. < 0.04%). They correspond to 83% of the non reactive aggregates, including 91% (10/11), 88% (7/8), 56% (5/9) and 100% (8/8) of the non reactive aggregates from the Trenton, Black River, Chazy and Beekmantown Groups, respectively (Table 3). Using Rc < 100 and Rc* < 9.5 as additional limit criterias to the Dissolved Silica values (Sc and Sc*) significantly ameliorate the efficiency of the Method to distinguish the non reactive aggregates, since only 50% of the non reactive aggregates produce Sc values < 100 mm/l or Sc* values < 10 mm/l. On the other hand, 71% (25/35) of the reactive aggregates tested produce Sc values > 300 **or** Sc* >40, this including 72% (23/32) of the expansive Trenton limestones. The 120 < Sc < 200 "grey-zone" delimited from the preliminary tests results (section 3.1.3) is thus extended to Sc values between 100 and 300 mm/l values (with Rc >100) (Figure 10). A grey-zone is also obtained for Sc* values between 10 and 40 mm/l (Rc* >9.5) (Figure 11). Globally, 3% (1/33) of the Trenton limestones tested with Sc >100, Rc >100, Sc* > 10 and Rc* > 9.5 are non expansive in the Concrete Prism Test, while this value is 25% (1/4), and 100% (4/4) for the Black River and Chazy Groups, respectively.

Figures 12 and 13 show that, for the aggregates tested, there is no good correlation between the amount of dissolved silica (corrected or not) and the amount of expansion measured in the Concrete Prism Expansion Test. This indicates that the nature and the amount of insoluble residues within the carbonate rocks tested are not the only parameter which govern their potential for deleterious expansion in concrete. Indeed, the values of Sc and Rc measure the instability in alkaline solution of the potentially alkali-silica reactive material present in these aggregates (in other words their insoluble residues), but factors like the permeability and the porosity of the carbonate rocks should play a major role in their expansive behavior in concrete.

TABLE 3: Proportions of samples associated to each of the geological groups investigated, and classified according to their behaviors in the Modified Chemical Method and the Concrete Prism Test.

Group	Trenton		Black River		Chazy	Beekman.	Total	
# samples	43		11		9	8	71	
Concrete Prism Test (exp. 0.04%)[1]	Reac. 32 (74%)	N. react. 11 (26%)	Reac. 3 (27%)	N. reac. 8 (73%)	N. reac. 9 (100%)	N. reac. 8 (100%)	Reac. 35 (49%)	N. reac. 36 (51%)
Sc or Rc <100 or Sc* < 10 or Rc* < 9.5	-- --	10 (91%)	-- --	7 (88%)	5 (56%)	8 (100%)	-- --	30 (83%)
Sc>300 mm/l (Rc > 100) or Sc*>40 (Rc* > 9.5)	23 (72%)	1 (9%)	2 (67%)	1 (12%)	1 (11%)	0 ---	25 (71%)	3 (8%)
300>Sc>100 (Rc > 100) + 40>Sc*>10 (Rc* > 9.5)	9 (28%)	0 ---	1 (33%)	0 ---	3 (33%)	0 ---	10 (29%)	3 (8%)

1. Samples classified according to the 6 month expansion values obtained in the Concrete Prism Test (section 2.2). (**Non Reactive**: Exp. < 0.04%, **Reactive**: Exp. > 0.04%) .

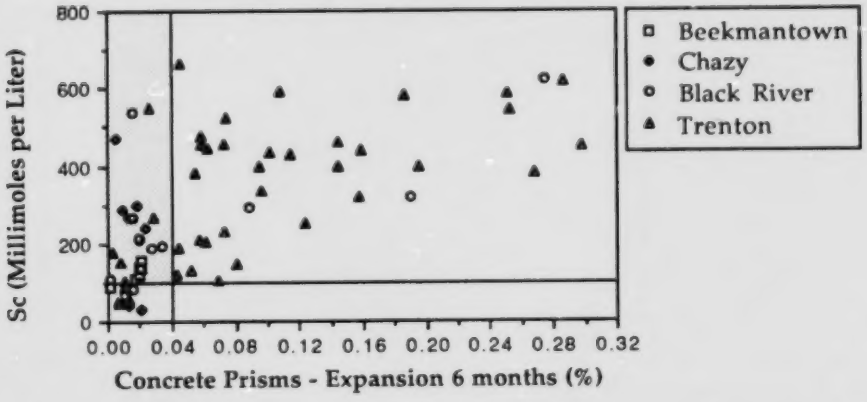


Figure 12: Sc values as a function of the 6 month expansion values measured in the Concrete Prism Test (Cement content = 350 kg/m^3). Samples are grouped according to their geological association.

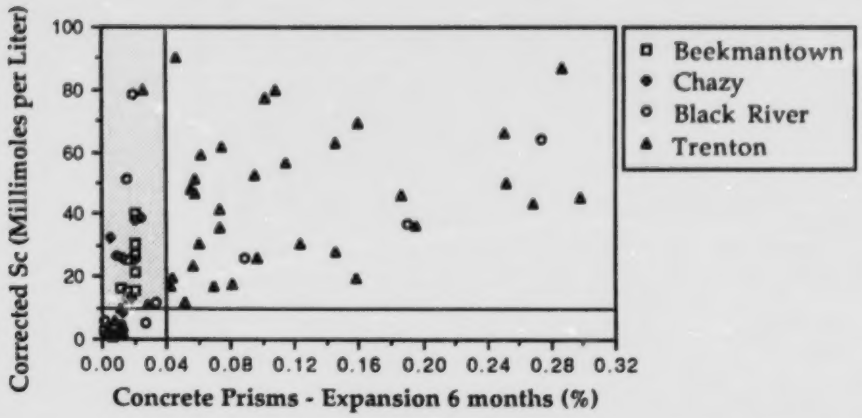


Figure 13: Corrected Sc values as a function of the 6 month expansion values measured in the Concrete Prism Test (cement content = 350 kg/m^3). Samples are grouped according to their geological association.

4. CONCLUSIONS

1. The Standard ASTM C289 Chemical Method is unreliable for the determination of the potential alkali-reactivity of carbonate aggregates exploited in the St.Lawrence Lowlands, since all the aggregates are classified innocuous. A modified version consisting of performing the chemical test on the insoluble residues of the aggregates is proposed.
2. Rc values are globally more influenced than the Sc values by the acid treatment and the particle size of the insoluble residues tested. Slightly higher Sc values are generally obtained when concentrated HCl (12N) is used for carbonate dissolution instead of diluted HCL (1.2N). Total insoluble residues (0-300 um) are more representative of the "potentially reactive material" present in each aggregate, and the best correlation with concrete prism expansion results is observed when they are obtained with concentrated HCl (12N).
3. The proposed test procedure thus involves acid treatment of unwashed 150-300 um bulk limestone or dolostone particles with concentrated HCl (12N). Total residues (0-300 um) are used to measure Sc values as described in the standard procedure ASTM C289. The percent of insoluble residues must be measured on each sample tested.
4. Relatively high coefficients of variation were calculated from the results obtained for 6 aggregates by six laboratories involved in an interlaboratory test program, and this, for each step of the investigated testing procedure (crushing efficiency, measurement of the percent of insoluble residues and Sc values). Coefficient of variations from 11% to 70% were obtained for the amount of dissolved silica depending upon the aggregate. It is probable that such variations should eventually induce the inadequate determination of the potential alkali-reactivity of some marginally reactive aggregates.
5. The Modified Chemical Method must be used concurrently with the petrographic examination of the carbonate aggregate under study. Information about the geological association of the rock (Formation or Member), and the composition and the nature of their insoluble residues are of great importance for the characterisation of their alkali-reactivity potential.
6. Among the 71 investigated aggregates from the St. Lawrence Lowlands, all the siliceous dolostones and sandy/dolomitic limestones from the Beekmantown, the Chazy, and the

Black River (Pamelia Formation) Groups give low expansions in the Concrete Prism Test (< 0.04% at 6 months), while all the reactive aggregates correspond to Black River or Trenton siliceous limestones. In addition, none of the investigated aggregates containing less than 5% insoluble residues produced excessive expansion at the Concrete Prism Test.

7. All the investigated aggregates meeting at least one of the following limit criterias: Sc < 100 mm/l, Rc <100 mm/l, Sc* < 10 mm/l, and Rc* < 9.5 mm/l (in the Modified Chemical Test) are non expansive aggregates. This correspond to 83% (30/36) of the non expansive aggregates recognized among the 71 aggregates tested. For the 41 remaining aggregates, only 3% (1/33) of those belonging to the Trenton Group are non expansive in the Concrete Prism Test, while this value increases to 25% (1/4) and 100% (4/4) for the Black River and Chazy Groups, respectively.

5. ACKNOWLEGMENTS

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6. REFERENCES

[1] Durand, M., Etude des propriétés physiques et chimiques de calcaires de la région de Montréal, Unpublished M.Sc. Thesis, Ecole Polytechnique de Montréal, 199p, 1969.

[2] Prudhomme, M., Caractérisation des granulats de carrières dans la région de Montréal. Unpublished M.Sc. Thesis, Ecole Polytechnique de Montréal, 103p, 1982.

[3] Bérard, J. & Roux, R., La viabilité des bétons du Québec: le rôle des granulats, Canadian Journal of Civil Engineering, 13, 12-24, 1986.

[4] Houde, R., Etude des granulats à béton réactifs aux alcalis de la région de Trois-Rivières, Unpublished M.Sc. Thesis, Ecole Polytechnique de Montréal, 188p, 1986.

[5] Bérubé, M.A. & Fournier, B. , Les produits de la réaction alcalis-silice dans le béton: Etude de cas de la région de Québec, Canadian Mineralogist, 24, 271-288, 1986 .

[6] Fournier, B., Bérubé, M.A. & Vézina, D., Investigation of the Alkali-Reactivity Potential of Limestone Aggregates from the Quebec City Area (Canada), Proceedings of the 7th International Conference on Alkali-Aggregate Reaction, Ottawa (Canada), Noyes Publication, P.E. Grattan-Bellew Editor, 23-29, 1987.

[7] Fournier, B., Bérubé, M.A. & Vézina, D., Condition Survey of Concrete Structures Built with Potentially Reactive Aggregates from the Quebec City Area, Proceedings of the Katherine and Bryant Mather International Conference on Concrete Durability, Atlanta (USA), ACI-SP-100, 1343-1364, 1987.

[8] Rogers, C.A., General Information on Standard Alkali-Reactive Aggregates from Ontario, Canada, Engineering Materials Office, Ministry of Transportation, Downsview, Ontario, 59p., 1988.

[9] Fournier, B. & Bérubé, M.A., Alkali-reactivity Potential of Carbonate Rocks from the St. Lawrence Lowlands (Quebec, Canada), Proceedings of the 8th International Conference on Alkali-Aggregate Reaction, Kyoto (Japan), K. Okada, S. Hishibayashi et M. Kawamura éditors, 363-368, 1989.

[10] ASTM C289 Standard Test Method for Potential Reactivity of Aggregates (Chemical Method), Annual Book of ASTM Standards, 04.02 Concrete and Mineral Aggregates, American Society for Testing and Materials, Race Street, Philadelphia, PA 19103, USA, 200-207, 1985.

[11] Fournier, B. & Bérubé, M.A., Caractérisation des granulats calcaires exploités dans la région de Québec et évaluation de leur potentiel de réactivité alcaline, Groupe de

Recherche en Géologie de l'Ingénieur, Report 85-20, University Laval (Québec), 207p., 1985.

[12] Mielenz, R.C. & Benton, E.J., Evaluation of the Quick Chemical Test for Alkali-Reactivity of Concrete Aggregate, HRB Bulletin, 171, 1-15, 1958.

[13] Bérubé, M.A. & Fournier, B., The Use of the Chemical Method ASTM C289 (on Insoluble Residues) and of the Accelerated South African Mortar Bar Test for Evaluating the Alkali-Reactivity Potential in Concrete of Siliceous Limestone Aggregates, Progress Report #88-22, Groupe de Recherche en Géologie de l'Ingénieur, University Laval, 45p. 1988.

[14] Globensky, Y., Géologie des Basses-Terres du St. Laurent. Ministère de l'Energie et des Ressources du Québec, Report MM 85-02, 63p. + map, 1987.

[15] Brun, J. & Chagnon, A., Rock Stratigraphy and Clay Mineralogy of Volcanic Ash Beds from the Black River and Trenton Groups (Middle Ordovician) of Southern Quebec, Canadian Journal of Earth Science, 16, 1499-1507, 1979.

[16] Brun, J. & Globensky, Y., Stratigraphy, Petrography, and Sedimentology of the Mohawkien of the Laurentian Platform, Joliette and Quebec Areas, 69th Reunion New-England Intercollegiate Geological Conference, Guidebook for field trips in the Quebec City Area, Excursion A-7, Béland and LaSalle Editors, 1977.

[17] Nissaire, M.L.T., Etude qualitative des calcaires dans les régions de Baie St-Paul-La-Malbaie, Simard et Havre-St.Pierre, DPV-788, Energie et Ressources Québec, 50p, 1982.

[18] Harland, T.L. & Pickerill, R.K., A review of Middle Ordovician Sedimentation in the St. Lawrence Lowland, Eastern Canada, Geol. Journal, 17, 135-156, 1982.

[19] Bezys, R.K. & Johnson, M.D., The Geology of the Paleozoic Formation utilized by the Limestone Industry of Ontario, CIM Bulletin, 81, 912, 49-58, 1988.

[20] Durand, B. & Bérard, J., Use of Gel Composition as a Criterion for Diagnosis of Alkali-Aggregate Reactivity in Concrete Containing Siliceous Limestone Aggregate. Materials and Structures / Matériaux et Construction, 20, 39-43, 1987.

[21] Morneau, L., Comparaison entre la Méthode Chimique ASTM C289 et un essai modifié - Etude des granulats calcaires face aux réactions alcalis-silice. Unpublished undergraduate project report, University Laval, 1987.

[22] Mielenz, R.C., Greene, K.T. & Benton, E.J., Chemical Test for Reactivity of Aggregates with Cement Alkalies - Chemical Processes in Cement-Aggregate Reaction, Journal of the American Concrete Institute, 19, 193-219, 1947.

[23] Vivian, H.E., A Working Appraisal of Alkali-Aggregate Reaction in Concrete, Proceedings of the 5th International Conference on Alkali-Aggregate Reactivity, Cape Town (South Africa), NBRI Editor, Paper S252/14, 1981.

[24] Thaulow, N. & Olafsson, H., Alkali-Silica Reactivity of Sands, Comparison of Various Test Methods. Proceedings of the 6th International Conference on Alkali-Aggregate Reactivity, Copenhagen (Denmark), G.M. Idorn and Rostam Editors, Danish Concrete Association, 359-364, 1983.

[25] Sims, I., Application of Standard Testing Proceedures for Alkali-Reactivity - Part 1, Concrete, 27-29, 1981.

[26] Grattan-Bellew, P.E., Evaluation of Test Methods for Alkali-Aggregate Reactivity, Proceedings of the 6th International Conference on Alkali-Aggregate Reactivity, Copenhagen (Denmark), G.M. Idorn and Rostam Editors, Danish Concrete Association, 303-314, 1983.

[27] Bérubé, M.A., CSA Interlaboratory Study on the Modified Chemical Method (on Insoluble Residues) Used to Evaluate the Potential Alkali-Silica Reactivity of Carbonate Aggregates, Progress Report, University Laval, 11p. 1989.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

INTERLABORATORY STUDY OF THE CONCRETE PRISM EXPANSION TEST FOR THE ALKALI-CARBONATE REACTION

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ABSTRACT

In Canada, the concrete prism expansion test is used for measuring expansion caused by alkali-carbonate reactive concrete aggregate. Three (3) concrete prisms about 75 x 75 x 400 mm, are made with the suspect aggregate and 310 kg/m^3 cement with an alkali content of 1.25% Na_2O equivalent. This report describes a study of the precision of this test sponsored by Canadian Standards Association Committees A5 and A23. Twenty laboratories participated in this study. The coarse aggregate was from the first lift of the Pittsburg quarry in Kingston, Ontario. Each laboratory made twelve (12) concrete prisms, which were stored in one of four different environments: moist room at 23°C, sealed in plastic bags with 100 ml of water in the moist room, immersed in 5 percent sodium chloride solution at 23°C, and over water in a sealed box at 38°C. The multi-laboratory variation was the same for all storage conditions. The average coefficient of variation of 23.4% was almost identical to that found in an earlier study. There were substantial differences in expansion found in the different storage conditions. Storage in the moist room gave the least expansion, probably due to alkali leaching.

INTRODUCTION

The alkali-carbonate rock reaction was first described by Swenson in 1957. This reaction can cause excessive expansion and cracking of Portland cement concrete within three years. It occurs with quarried sedimentary dolomitic limestones with moderate to high clay contents. Typical expansive carbonates of this type, in Ontario, have CaO:MgO ratios of 5 to 10, alumina (Al_2O_3) contents of 2 to 3%, and insoluble residues of 10 to 15% (Rogers, 1986). Higher cement alkali contents result in increased expansion. The pessimum proportion is 100%. Expansion of concrete is caused by swelling of individual coarse aggregate particles. This is thought to be due to adsorption of water on previously dry clay surfaces in the rock (Gillott, 1975).

There are four procedures for testing aggregate for this reaction:

1. Petrographic examination of thin sections of the rock, to observe the characteristic reactive texture of euhedral dolomite rhombs in a clayey, micritic matrix.

2. The rock-cylinder expansion test that measures expansion when stored in alkali solutions (ASTM C 586).

3. A quick chemical test, used in Ontario, that identifies those aggregates that have a bulk composition corresponding to known reactive carbonate aggregates (Rogers, 1986).

4. The concrete prism expansion test, normally used as the specification method to measure expansion of concrete containing aggregate recognized as being potentially reactive using the other tests (CSA A23.2-14A).

CONCRETE PRISM EXPANSION TEST

In early studies of the alkali-carbonate reaction, the mortar bar expansion test gave insufficient expansion (Swenson, 1957). Swenson developed a concrete prism expansion test that has been adopted by the Canadian Standards Association (CAN A23.2-14A). Three concrete prisms (75 x 75 x 350 mm to 120 x 120 x 450 mm) are made using the suspect coarse aggregate in a mixture with 310 kg/m^3 of normal Portland cement and a non-reactive fine aggregate. The alkali content of the cement should normally be 1.0 ± 0.2% Na_2O. Sodium hydroxide is added to the mixture to give a final equivalent alkali content of 1.25% Na_2O by mass of cement or the highest cement alkali content likely to be encountered. On demoulding, the prisms are stored in a moist room and expansion measured periodically and reported as a percent change in initial length. The average expansion of the three prisms is reported as the final test result.

The 1986 Canadian Standard recommended that expansions in excess of 0.01% at 3 months or, failing that, expansions in excess of 0.025% at 1 year were suggested maxima for concrete exposed to moist conditions with sodium chloride present.

An earlier multi-laboratory study (Rogers, 1987) found that there was a considerable laboratory bias in this test. For expansions greater than 0.014%, the results of properly conducted tests on the same aggregate by two laboratories should not differ by more than 65% of the average. This variation was the same even when the same fine and coarse aggregate, cement and mix design were used by the different laboratories. It was concluded that one of the major sources of variation was the conditions of moist storage of the concrete prisms. Rogers and Hooton (1989) stored concrete prisms under different storage conditions. They found that the prisms showing the least expansion had lost the greatest amount of cement alkalies due to leaching. Prisms

stored in the normal moist curing room at 23°C lost the greatest amount of alkalies. Storing the prisms in either plastic bags with 100 ml of water or immersed in 5% sodium chloride solution reduced or eliminated alkali leaching and resulted in more expansion of the concrete.

PURPOSE OF STUDY

The primary purpose of this study was to gain data to formulate a multi-laboratory precision statement over a range of expansion values for the concrete prism expansion test. A secondary purpose was to try and find a storage condition that gave less multi-laboratory variation than the conventional storage in the moist curing room at 23°C.

Design

The ASTM Practice for conducting interlaboratory test programs to determine the precision of test methods was followed as far as possible (ASTM C 802). ASTM recommends that, for 15 participating laboratories, at least two replicates of each material are tested by each laboratory so that n is equal to or greater than 30. In this study, such a requirement would have resulted in an unacceptable amount of work and high cost. The ASTM practice recommends a minimum of three materials. In this test, there is more than one test result for each prism. Expansion results are obtained at various ages. Expansion at each age is amenable to statistical treatment for calculation of variance. It was decided that the large amount of data at different ages would compensate for not testing replicate materials. This design also precluded estimates of within-laboratory precision.

The study design was as follows:

Laboratories were asked to make twelve prisms of concrete according to CSA A23.2-14A. A known alkali-carbonate reactive coarse aggregate was supplied for the study. Each laboratory used its own fine aggregate and cement meeting the requirements of the test procedure. The laboratories were required, after demoulding and initial length measurement, to store the prisms in one of four environments:

1. In the moist curing room at 23 ± 2°C (as for the test procedure);

2. As 1 above, but each prism was sealed in a plastic bag with 100 ml of tap water. The tap water was renewed and the bag resealed after every measurement;

3. Immersed in 5 percent sodium chloride solution in a container with a lid to prevent evaporation;

4. Over water in a sealed box (Figure 1) with filter paper wicks in a room or container at 38°C. A standard plastic box was supplied by the co-ordinating laboratory.

The laboratories were instructed to randomly assign three prisms to each storage condition and measure length of each for two years.

Laboratories

Twenty experienced concrete testing laboratories participated, two in the U.S.A., one in France and seventeen in Canada. The laboratories are designated by the numbers 1 to 20. Laboratories 4, 9, 10, 13, 14, 17, 18 and 19 had no or limited experience with this procedure. The other laboratories had been conducting this test for many years.

Coarse Aggregate

The coarse aggregate was a 4.75-19 mm quarried dolomitic limestone from the first lift of the Pittsburg quarry, near Kingston, Ontario. This stone is well known as being alkali-carbonate reactive (Rogers, 1986).

One hundred and twenty canvas bags, each of about 30 kg capacity were filled from a 100 tonne stockpile at the Ministry of Transportation, Ontario. The bags were taken to the laboratory, the contents oven dried and the bags assigned randomly to each of the laboratories. No attempt was made to grade or split the bulk material, the reason being, that this rarely occurred in normal practice. "Precision estimates for inclusion in a test method must be obtained under conditions... that are representative of the situations in which the test method will be used in practice" (ASTM C 802-80). Five bags were sent to each laboratory.

Extra bags were kept for supplying laboratories in the event of the need for more aggregate. Two laboratories (17, 19) had to request more aggregate for re-testing because of unexplained problems in measurement.

RESULTS AND DISCUSSION

Expansion data from 28-168 days are shown in Tables 1-5. A number of laboratories' data was omitted from analysis. Laboratory 5 used two different measuring apparatus. The data labelled 5.1 was obtained with apparatus (Swenson type) illustrated in the 1986 version of the procedure. The data labelled 5.2 was obtained by measuring the same prisms as used for 5.1 with an apparatus (mortar bar type) similar to, but larger than, that illustrated in ASTM C 490. This device allows the prism to be slowly rotated to obtain a minimum, but consistent, reading. Laboratory 5 had found, in an earlier study, that the mortar bar device gave half as much variation between the three individual prisms as given by measuring the same prisms with the Swenson Type device. Both results are presented but only those obtained with the mortar bar device (5.2) were used in statistical calculation.

In a few cases, individual measurements were excluded from statistical analysis because they were very different from the other data. Examples are: Table 3, lab 19, 5% NaCl; Table 5, lab 19, 5% NaCl. Laboratory 19 had no previous experience with this test and had to abandon a previous concrete prism test in this program due to unexplained problems. Data from laboratory 4 was also excluded at later ages. This laboratory noted that its expansion data, especially at 38°C was much greater than others. Mortar bar tests showed that the fine aggregate used was probably alkali-silica reactive. The alkali-silica expansive nature of the fine aggregate increased the rate of expansion of the concrete prisms.

Multi-laboratory Precision

Following the rejection of outlying data, the coefficient of variation at each age, for each storage condition was plotted against expansion (Figure 2). It can be seen that there is no apparent influence of storage condition on multi-laboratory variation. The average coefficient of variation for all storage conditions was 23.4%. This agrees with the average multi-laboratory coefficient of variation of 23% for expansion greater than 0.014% found in the earlier study (Rogers, 1987). The lack of change in coefficient of variation with change in storage condition demonstrates that there must be some other cause of the wide multi-laboratory variability of this test. In the earlier study (Rogers, 1987) it was shown that variation was not reduced when all laboratories used the same fine aggregate, cement and mix design. The major source of variation remaining to be investigated is the measurement process. This is not to say that the nature of the fine aggregate, or any other variable, is not a potential source of variation. The example of laboratory 4, in this study, is sufficient evidence.

Storage Conditions

Storage environment also has a major influence on expansion, as shown in Figure 3. Rogers and Hooton (1989) showed that concrete prisms stored in these different environments showed significant variation in the amount of alkalies leached from the concrete. the concrete prisms stored in the conventional moist room had lost the greatest amount of the alkalies (63%) after 2½ years. Prisms stored in plastic bags only lost 22% of their alkali. Rogers and Hooton attributed these variations in expansion to leaching of the alkalies. Because of the removal of alkalies, the alkali- aggregate reaction will slow down or cease in conditions where there is continual source of fresh water falling on the concrete, removing alkali. The concrete in the plastic bags is also stored in 100% humidity but it does not have a constant source of fresh water to remove alkali. The water in the plastic bag approaches equilibrium, with respect to alkali concentration, with that of the cement paste solution inside the prism.

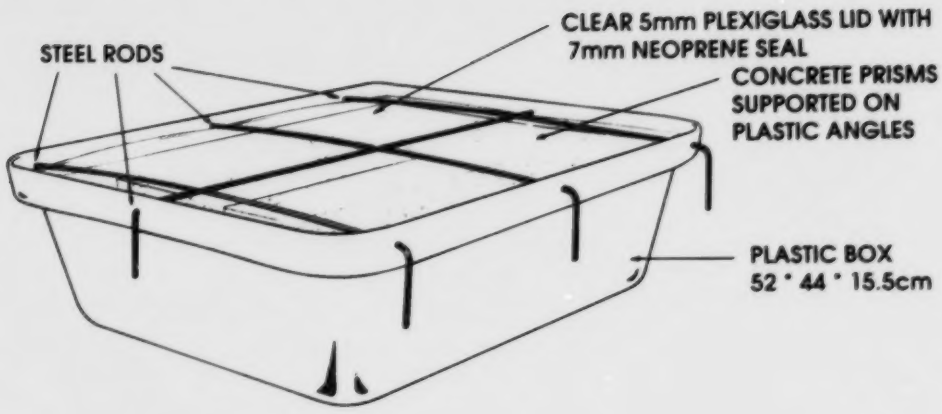

FIGURE 1 - Container used by all laboratories for storage of concrete at 38° C, over water.

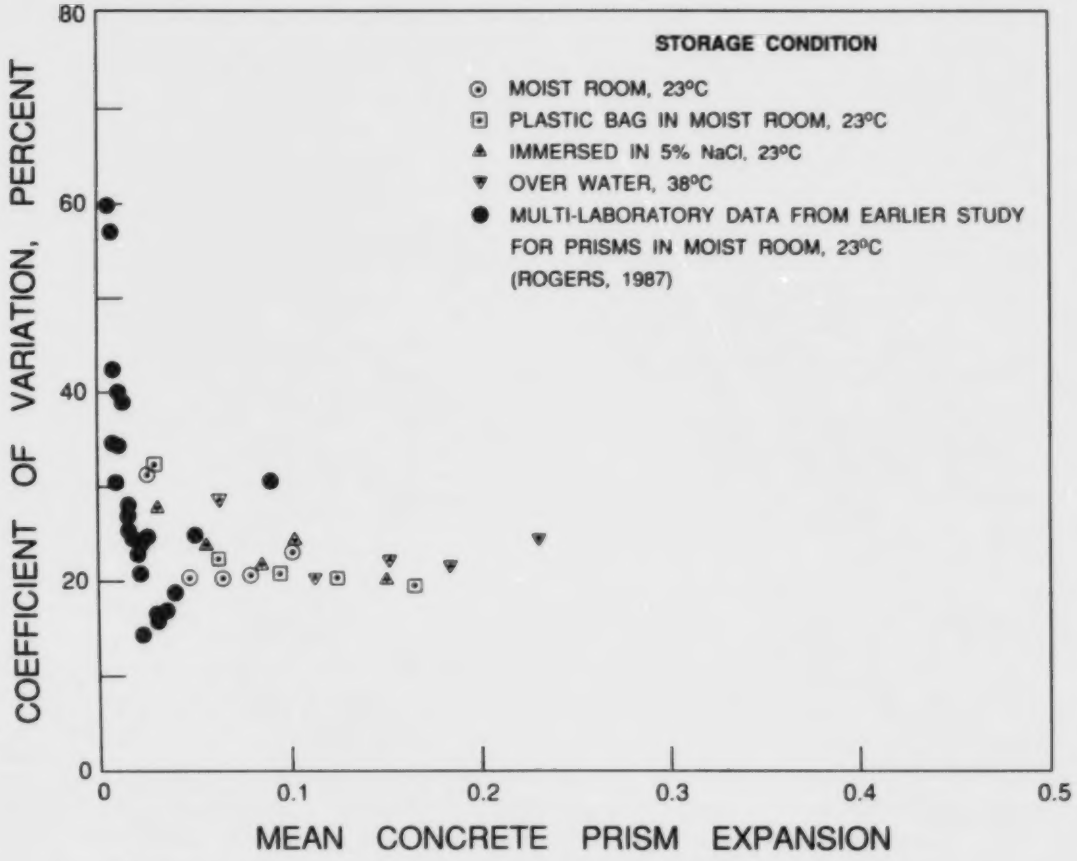

FIGURE 2 - Coefficient of variation against mean expansion under various storage conditions.

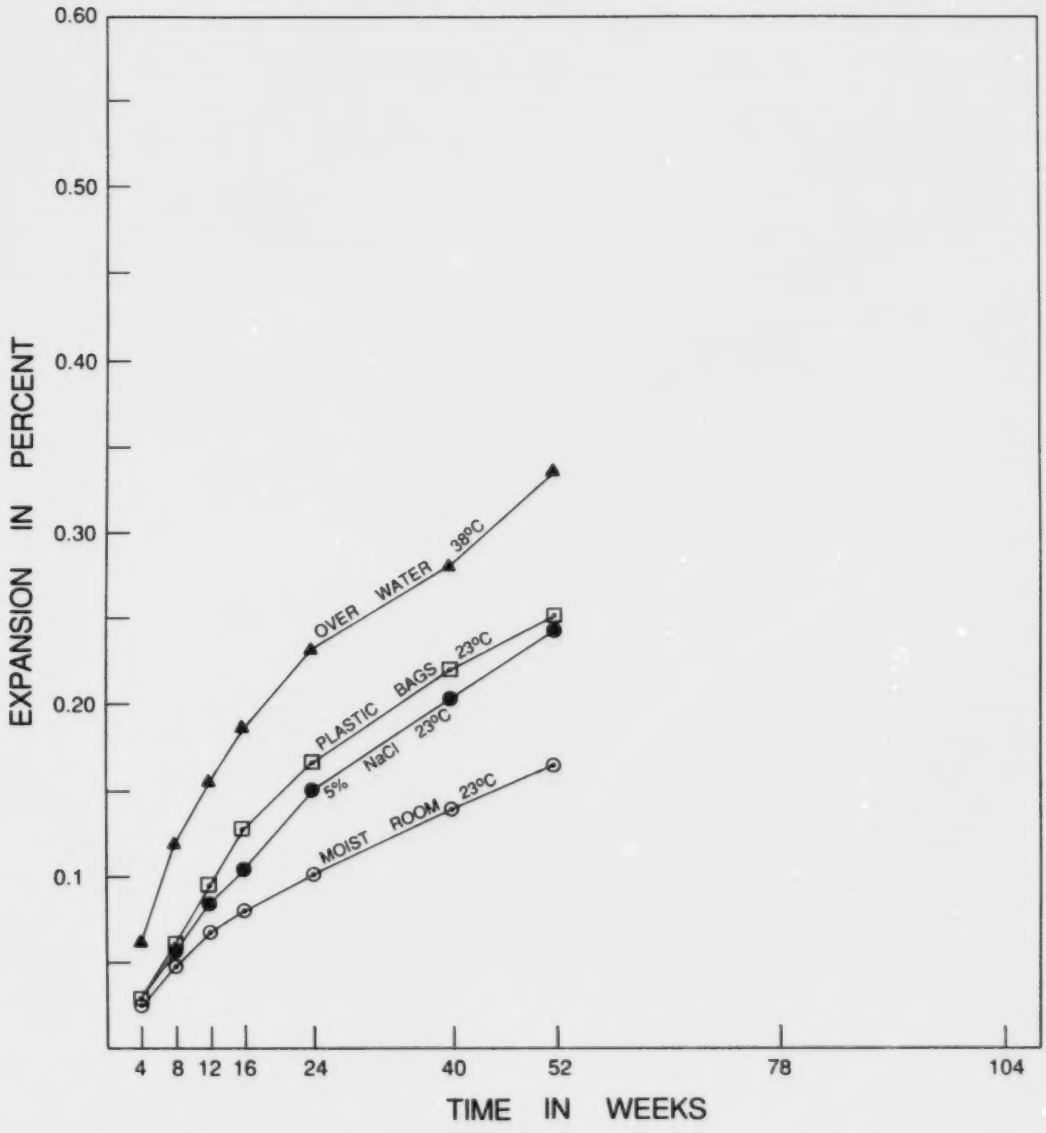

FIGURE 3 - Average concrete prism expansion against time for various storage conditions.

Which of the laboratory storage environments is most representative of the conditions that are found in practice? Clearly, concrete stored unprotected in the moist room shows the least expansion, yet this has been considered a severe environment by many concrete engineers. The Ministry of Transportation in Ontario has been conducting a comparison of field expansion of concrete sidewalks with expansion of concrete under various laboratory conditions. After four years, the best correlation with field expansion was that obtained with concrete prisms stored in 5% sodium chloride solution at 23°C.

CONCLUSIONS

1. Multi-laboratory study of the concrete prism expansion test showed that the coefficient of variation was 23.4%. This agrees well with an earlier study reported in 1987. Therefore, results of two properly conducted tests in different laboratories on the same aggregate should not differ from each other by more than about 66 percent of their average.

2. Different storage conditions caused significantly different expansion of concrete. It is postulated that these different expansions are related to variable leaching of alkalies from the concrete. Concrete stored unprotected in the moist curing room gave the least average expansion.

3. The multi-laboratory variation was not changed by using concrete prism storage conditions different from the conventional moist curing room.

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The cheerful participation of these laboratories and the good work of their staff is eloquent testimony to the interest in concrete durability in North America.

REFERENCES

ASTM C 586 - Standard Test Method for Potential Alkali-Reactivity of Carbonate Rocks for Concrete Aggregates (Rock Cylinder Method); American Society for Testing and Materials, Annual Book of Standards, Vol.404.02.

ASTM C 670 - Standard Practice for Preparing Precision Statements for Test Methods for Construction Materials; American Society for Testing and Materials, Annual Book of Standards, Vo.04.02.

ASTM C 802 - Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials; American Society for Testing and Materials, Annual Book of Standards, Vol.04.02.

CAN3-A23.1-M77: 1986; Concrete Materials and Methods of Concrete Construction - Appendix B, Alkali Aggregate Reaction. Supplement No. 2; Canadian Standards Association, Rexdale, Ontario.

CAN3-A23.2-14A: 1986; Test for Alkali-Aggregate Reaction; Methods of Test for Concrete, Supplement No.2, Canadian Standards Association, Rexdale, Ontario.

Gillott, J.E.; 1975; Alkali-Aggregate Reactions in Concrete; Engineering Geology, Vol.9, pp.303-326.

Rogers, C.A.; 1986; Evaluation of the Potential for Expansion & Cracking of concrete due to the Alkali-Carbonate Reaction; Cement, Concrete and Aggregates, CCAGDP, Vol.8, No.1, Summer 1986, pp.13-23.

Rogers, C.A.; 1987; Interlaboratory study of the concrete prism expansion test for the alkali-carbonate reaction; Ontario Ministry of Transportation, Engineering Materials Office, Report EM-76, 35p.

Rogers, C.A.; R.D. Hooton; 1989; Leaching of alkalies in alkali-aggregate reaction testing; Proceedings 8th International Conference on Alkali-Aggregate Reaction, Kyoto, Japan, pp.327-332.

Swenson, E.G.; 1957; A Reactive Aggregate Undetected by ASTM Tests; ASTM Bulletin, No.226, Dec., pp.48-51.

TABLE 1 - Results Obtained at 28 days

Laboratory No.	Moist Room 23°C	Plastic Bags in Moist Room 23°C	5% NaCl Solution 23°C	Over Water 38°C
1	0.0070	0.0210	0.0257	0.0665
2	0.0284	0.0378	0.0376	-
3	0.0325	0.0379	0.0399	0.0770
4	0.0223	0.0287	0.0233	0.0830
5.1 (2)	0.0275	0.0442	0.0323	0.0441
5.2	0.0365	0.0467	0.0372	0.0548
6	0.0213	0.0265	0.0222	0.0585
7	0.0250	0.0245	0.0287	0.0338
8	0.0389	0.0422	0.0386	0.0758
9	0.0230	0.0273	0.0257	0.0867
10	0.0197	0.0243	0.0265	0.0197
11	0.0254	0.0344	0.0361	0.0852
12	0.0307	0.0270	0.0240	0.0593
13	0.0230	0.0233	0.0193	0.0617
14	0.0239	0.0293	0.0325	0.0694
15	0.0107	0.0073	0.0127	0.0630
16	0.0323	0.0330	0.0340	0.0480
17	0.0302	0.0328	0.0391	0.0754
18 (1)	0.0338	0.0136	0.0374	0.0652
19	0.0234	0.0319	0.0192	0.0439
20				
Mean	0.02568	0.02892	0.02946	0.06261
6-1	0.008042	0.009346	0.008152	0.017894
Coef. of Var.	31.3%	32.3%	27.7%	28.6%
n	19	19	19	19

NOTE: 1. Data for average of 2 prisms.
2. Lab 5.1 excluded in all calculations.

TABLE 2 - Results Obtained at 56 days

Laboratory No.	Moist Room 23°C	Plastic Bags in Moist Room 23°C	5% NaCl Solution 23°C	Over Water 38°C
1	0.0385	0.0572	0.0525	0.1167
2	0.0446	0.0700	0.0657	-
3	0.0640	0.0810	0.0772	0.1331
4	0.0407	0.0677	0.0523	0.1860*
5.1 (2)	0.0452	0.0854	0.0637	0.1010
5.2	0.0702	0.0843	0.0636	0.1150
6	0.0492	0.0594	0.0513	0.1251
7	0.0426	0.0435	0.0552	0.0678
8	0.0677	0.0880	0.0736	0.1310
9 (59d)	0.0430	0.0653	0.0490	0.1560
10 (61d)	0.0527	0.0720	0.0617	0.1027
11	0.0377	0.0598	0.0593	0.1457
12	0.0463	0.0533	0.0446	0.0896
13	0.0460	0.0600	0.0400	0.1090
14	0.0406	0.0583	0.0608	0.1261
15	0.0393	0.0357	0.0320	0.1217
16	0.0450	0.0577	0.0627	0.0803
17	0.0564	0.0697	0.0734	0.1223
18 (1)	0.0496	0.0397	0.0525	0.1101
19	0.0394	0.0593	0.0287	0.0857
20				
Mean	0.04808	0.06221	0.05558	0.11399
6-1	0.009914	0.013915	0.013206	0.023269
Coef. of Var.	20.6%	22.4%	23.8%	20.4%
n	19	19	19	17
Outlier	-	-	-	4

NOTE: 1. Data for average of 2 prisms.
2. Lab 5.1 excluded in all calculations.

TABLE 3 - Results Obtained at 84 days

Laboratory No.	Moist Room 23°C	Plastic Bags in Moist Room 23°C	5% NaCl Solution 23°C	Over Water 38°C
1	0.0618	0.1003	0.0712	0.1492
2	0.0583	0.1006	0.0971	-
3	0.0896	0.1204	0.1130	0.1732
4	0.0620	0.1180	0.0940	0.2720*
5.1 (2)	0.0668	0.1247	0.0960	0.1157
5.2	0.0873	0.1210	0.0950	0.1326
6	0.0612	0.0916	0.0744	0.1865
7	0.0579	0.0617	0.0792	0.0921
8	0.0911	0.1296	0.1074	0.1702
9 (59d)	0.0523	0.1010	0.0700	0.1987
10 (61d)	0.0633	0.1037	0.1240	0.1803
11	0.0472	0.0863	0.0814	0.2085
12	0.0580	0.0830	0.0730	0.1213
13	0.0690	0.0963	0.0703	0.1443
14	0.0561	0.0874	0.0914	0.1716
15	0.0573	0.0606	0.0510	0.1570
16	0.0560	0.0807	0.0877	0.1077
17	0.0821	0.1072	0.1066	0.1617
18 (1)	0.0669	0.0659	0.0651	0.1149
19	0.0516	0.0870	0.0347*	0.1164
20				
Mean	0.06468	0.09486	0.08621	0.15213
6-1	0.013233	0.019812	0.018957	0.033753
Coef. of Var.	20.5%	20.9%	22.0%	22.2%
n	19	19	18	17
Outlier	-	-	19	4

NOTE: 1. Data for average of 2 prisms.
2. Lab 5.1 excluded in all calculations.

TABLE 4 - Results Obtained at 112 days

Laboratory No.	Moist Room 23°C	Plastic Bags in Moist Room 23°C	5% NaCl Solution 23°C	Over Water 38°C
1	0.0840	0.1178	0.1050	0.1807
2	0.0702	0.1307	0.1255	-
3	0.1085	0.1516	0.1349	0.2001
4	0.0740	0.1737	0.1580*	0.3380*
5.1 (2)	0.0756	0.1443	0.1068	0.1226
5.2	0.0749	0.1418	0.1112	0.1400
6	0.0819	0.1289	0.0931	0.2490
7				
8	0.1160	0.1707	0.1432	0.2010
9 (111d)	0.0597	0.1343	0.0853	0.2300
10	0.0953	0.1523	0.1240	0.1803
11	0.0543	0.1061	0.0968	0.2572
12	0.0697	0.1093	0.0930	0.1420
13	0.0853	0.1278	0.0907	0.1692
14	0.0709	0.1151	0.1242	0.2099
15	0.0780	0.0883	0.0696	0.1966
16	0.0720	0.1087	0.1137	0.1310
17	0.1031	0.1304	0.1314	0.1905
18 (1)	0.0743	0.0814	0.0719	0.1322
19	0.0661	0.1022	0.0528	0.1428
20				
Mean	0.07990	0.12617	0.10390	0.18453
6-1	0.016515	0.025586	0.025388	0.040206
Coef. of Var.	20.7%	20.3%	24.4%	21.8%
n	18	18	17	16
Outlier	-	-	4	4

NOTE: 1. Data for average of 2 prisms.
2. Lab 5.1 excluded in all calculations.

TABLE 5 - Results Obtained at 168 days

Laboratory No.	Moist Room 23°C	Plastic Bags in Moist Room 23°C	5% NaCl Solution 23°C	Over Water 38°C
1	0.1097	0.1551	0.1446	0.2262
2	0.0924	0.1741	0.1790	-
3	0.1501	0.2116	0.1892	0.2512
4	0.0960*	0.2620*	0.3260*	0.4330*
5.1 (2)	0.1021	0.1875	0.1558	0.1627
5.2	0.0951	0.1796	0.1572	0.1694
6 (203 d)	0.1201	0.1931	0.1572	0.3691
7				
8	0.1460	0.2185	0.1979	0.2329
9 (171 d)	0.0830	0.2123	0.1310	0.2777
10	0.1190	0.1943	0.1660	0.2600
11	0.0593	0.1268	0.1072	0.2923
12	0.0767	0.1386	0.1326	0.1690
13	0.1126	0.1761	0.1315	0.2047
14	0.0941	0.1544	0.1794	0.2660
15	0.1056	0.1286	0.1070	0.2413
16	0.0870	0.1457	0.1570	0.1743
17	0.1257	0.1638	0.1720	0.2341
18 (1)	0.0971	0.1185	0.0953	0.1514
19	0.0855	0.1311	0.0663*	0.1802
20				
Mean	0.10347	0.16601	0.15026	0.23124
6-1	0.023879	0.032307	0.030699	0.056620
Coef. of Var.	23.1%	19.5%	20.4%	24.5%
M	17	17	16	16
Outlier	4	4	4, 19	4

NOTE: 1. Data for average of 2 prisms.
2. Lab 5.1 excluded in all calculations.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

ALKALI-AGGREGATE REACTIVITY IN CANADA FROM A CEMENT INDUSTRY PERSPECTIVE

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1. INTRODUCTION

Research and testing in the past 30 years has shown that, in several regions of Canada, alkali-aggregate reactivity (AAR) presents problems. The fact that the 7th International Conference on AAR was held in Ottawa in August, 1986, was not a random choice of location. Some 200 people from 23 countries were present at the Conference, which indicates that this problem is worldwide and not unique to Canada.[1]

In this paper I will attempt to give a brief overview of:

- The types of problems encountered and their geographical incidence;
- The current requirements of CSA Standards;
- Control measures in place and research;
- Cement industry concerns; and
- Initiatives which have been taken by CPCA towards eventually providing concrete users with assurance that AAR problems will not occur.

2. AAR PROBLEMS, THEIR CAUSE AND INCIDENCE IN CANADA

Alkali-aggregate reactivity is a type of concrete deterioration which occurs due to a reaction between the

active mineral constituents of some aggregates and the alkaline components in the concrete mix, which are usually derived from the cement.

All natural rocks react to some extent and, generally, this reaction is beneficial, in that it creates good bond between the cement paste and the aggregate and with reinforcing steel. In some instances, however, the reaction can be harmful or deleterious, in that it results in excessive expansion and cracking of concrete exposed to moisture.

This cracking is undesirable for a number of reasons. It reduces the strength of the concrete. Cracks act as channels for water movement in concrete, leading to increased saturation and reduced freeze-thaw durability. This water movement results in leaching of cement paste, often seen as white deposits on the surface of the concrete. Cracks also lead to increased concentration of deicer salts in the concrete which accelerates deterioration. Thus, the actual expansion and cracking associated with AAR may not, in itself, be the main reason for concern. The major deterioration is often caused by secondary processes which occur after concrete cracking has been initiated by AAR.

Problems due to AAR may occur in concrete which is subject to a moist environment. Dams, bridge structures and paving are prime examples of the type of construction where such deleterious reactivity has been observed.

The reasons why AAR problems appear to be on the increase worldwide include:

- Depletion of good aggregate sources is resulting in the use of aggregates formerly considered to be of marginal quality;

- Increases in cement alkalis, in some areas, due to changes in manufacturing processes. These changes have been brought about by the national need to conserve energy and, at the same time, to reduce environmental pollution;

- Consumer pressures for higher early concrete strengths have resulted in both more finely ground cements and higher overall cement contents in concrete mixes. Both of these factors, together with high temperature steam curing of precast products, increase the potential for AAR; and

- The application of alkaline salts to concrete, due to the use of deicing chemicals, increases the expansion of concrete containing reactive aggregates. This increased

expansion could cause cracking and accelerated deterioration in concrete which might otherwise give satisfactory performance.

It should also be observed that increased awareness of AAR and improved methods for its detection now enable the identification of the problem in situations where cracking would previously have been attributed to other causes. Cracking of the parapets of the Prince Edward Viaduct in Toronto, which was built in 1918, is an example of this type of situation.[2]

Three types of AAR are encountered in Canada.[3] They are:

(i) **Alkali-silica reaction (ASR)**

Time/expansion graphs of concrete prisms or mortar bars made with ASR aggregates are characterized by an early onset of expansion and usually by a high rate of expansion. Cracking of structures suffering from ASR is usually observed within 10 years of construction.

(ii) **Slow/late expanding alkali silicate/silica reaction**

This type of reactivity is distinguished from ASR by the delayed onset of expansion of concrete test prisms and the length of time before cracking becomes evident in concrete structures, which may be up to 20 years.

(iii) **Alkali-carbonate reaction (ACR)**

Fortunately, to this time, the only known incidences of this type of AAR are confined to a couple of areas in Canada. Alkali-carbonate reactivity causes expansion and extensive cracking of concrete. Under laboratory conditions, it is usually characterized by the rapid onset of expansion of concrete test prisms. Structures undergoing this reaction usually show cracking within 5 years of construction.

Geographically, the incidence of AAR in Canada varies widely, as does the level of cement alkalis, expressed as Na_2O equivalent.[3]

- In British Columbia, cement alkalis are generally below 0.6%, because of the natural availability of low-alkali raw materials. Some potentially reactive aggregates are known to occur, but AAR problems are minimal.[4]

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- In the Prairie Provinces, cement alkalis are higher than in B.C., but are still relatively low. Some potentially reactive aggregates are, however, known to occur in some localized areas. Alkali-reactivity is generally a minimal problem.[4]

- In Ontario and Quebec, reactive aggregates are widespread. Cement alkalis are generally high, in the Ø.6 to greater than 1% range. AAR problems occur in several regions.

- In the Atlantic Provinces, AAR problems involving siliceous aggregates have been identified. Cement alkalis are moderately high.

3. REQUIREMENTS OF CSA CONCRETE STANDARDS

Since 196Ø, CSA Standard A23.1, "Concrete Materials and Methods of Concrete Construction",[3] which is referenced in most specifications, has included requirements to the effect that both coarse and fine aggregates for use in concrete shall not react with alkalis in the cement to an extent that may result in excessive expansion in the concrete. Reactive aggregates causing excessive expansion shall only be used when corrective measures acceptable to the Owner are applied.

The Standard then refers to its Appendix 'B', "Alkali-Aggregate Reaction", for a discussion of methods of evaluating potential reactivity and the application of corrective measures.

The provisions of the Appendix 'B' to the A23.1 Standard were extensively revised in 1986. The Appendix discusses the type and incidence of reactive aggregates in Canada and specifies and discusses the applicability of the test methods to be used to evaluate their potential reactivity. It provides detailed "flow-chart" information for the evaluation of aggregates and recommends limits of linear expansion under test which indicate when expansion should be considered to be deleterious.

While the Standard observes that probably the best means of evaluating the acceptability of aggregates is a field investigation of structures containing the aggregate, it notes that such structures should be at least 1Ø years old and be constructed of concrete having the same or higher level of cement alkalis, with aggregate from the same horizon or lift of the pit or quarry under consideration. Also, that the

existing structure should have been exposed to the same curing and environmental conditions as will be experienced by the proposed structure. Because of the probability that several of these factors will be unknown or uncertain, it can readily be seen that aggregate acceptance based solely on proven field performance is not to be recommended. Nonetheless, such a field investigation should, where possible, be carried out, as it can provide very useful evidence to assist in confirming the results of laboratory testing.

Appendix 'B' to CSA A23.1 also discusses the application of corrective measures which may, in some circumstances, be effective in minimizing or preventing the deleterious expansion and cracking of concrete due to AAR. The corrective measures suggested are:

- Selective extraction of aggregates to reduce or eliminate the reactive material; or
- Reduction of the cement content in the concrete mix or use of a cement with a low-alkali content or both; or
- Use of supplementary cementing materials.

The Standard goes on to note that application of any of these measures, other than selective aggregate extraction, depends on establishing proof of their effectiveness, generally by means of laboratory testing.

In another section of the A23.1 Standard, concrete suppliers are required to certify that all of the materials used in their concrete comply with the requirements of the Standard. Thus, legally speaking, the concrete supplier could be found to be liable if the extensive aggregate evaluation prescribed in Appendix 'B' of the Standard has not been carried out and the concrete develops alkali-aggregate reactivity problems.

It can be seen, therefore, that the current A23.1 Standard positively requires the evaluation of aggregates from the standpoint of alkali-aggregate reactivity. The Standard does not assign responsibility for making this evaluation, because this is considered to be an administrative requirement.

In practice, for most work, little if any such formal evaluation has been made in the past, with specification agencies relying on an assumption of good proven field performance. In areas where the problem has been identified,

this situation is likely to change radically in the future with increased awareness of AAR.

4. CONTROL MEASURES IN PLACE AND RESEARCH

To date, the Ontario Ministry of Transportation is the only major specifier to have established a formal program for the general evaluation of aggregate sources from the standpoint of AAR within the Province for MTO concrete construction. MTO publishes a list of approved aggregate sources for each region of Ontario, based on the results of an ongoing sampling and testing program.[2] For a source to be included on this list, it either has to have passed all applicable tests or have a proven record of past field performance in highway structures. The aggregate source is placed on the list, together with any restrictions. For instance, in some quarries, an approved stratum is specified since other levels may be alkali reactive.

The other main government effort in this area at present is administered by CANMET, which is a part of Energy, Mines and Resources Canada. This is carried out through Mineral Development Agreements (MDA's) which exist between the Federal Government and the Provinces. MDA work in the AAR area is presently underway in New Brunswick, Newfoundland and Nova Scotia. MDA work, the funding for which is 80% provided federally and 20% provincially, is generally aimed at providing information having a broad scientific scope. For example, in New Brunswick, the aim of the current project is to provide a generic identification of potentially deleterious native rock types and stops short of either mapping areas where these are likely to be found or identifying specific problem pits and quarries. The project in Newfoundland, which is directed towards ensuring that the proposed concrete offshore oil production structure for Hibernia will not contain deleteriously reactive aggregate, however, is aimed at identification of specific acceptable aggregate sources.

In addition to providing very substantial funding for the MDA programs, the Federal Government has also recently sponsored two significant programs of research in the AAR area. The first of these was into the influence of supplementary cementing materials on AAR.[5] It has long been known that use of pozzolanic mineral admixtures, now more commonly called "supplementary cementing materials", can be effective in reducing expansion due to AAR. The research program studied the relative effectiveness of 15 supplementary

cementing materials available in Canada on reducing the expansion of concretes made with each of the three recognised types of reactive aggregates. The results of this program confirm that supplementary cementing materials can be very useful in reducing the effects of some types of deleterious AAR and provides research information on the quantities of such materials required for various situations.

The second Federal Government-sponsored research program studied the effect of cement composition on AAR.[6] This extensive program, which involved making 12 laboratory cements and testing these, together with five other commercially available cements, investigated the effects of various levels of C_3S, C_2S, C_3A and C_4AF, free CaO and free MgO, as well as total cement alkalies on AAR. This program has confirmed that total cement alkalies play the predominant role, though other compositional characteristics may also have important effects.

A particular long-standing problem exists with many of the currently accepted methods of test for AAR, which is that six months to a year, or even two years, may be needed before conclusive results are obtained. These methods are also expensive. Work is going on, sponsored by several agencies including the Ontario Ministry of Transportation, Ontario Hydro, several Canadian universities, the CNR and PCA, towards the development of quick, inexpensive and reliable test methods. It now appears likely that several such methods will be available within the forseeable future.

5. CEMENT INDUSTRY CONCERNS AND INITIATIVES

From the standpoint of the cement and concrete industry, the corrective measure for AAR which, in many regions, causes the most problem is the specification of low or reduced alkali cement.

In cement manufacturing, the ability to produce a low-alkali cement depends primarily on the availability of low-alkali raw materials, with a secondary dependency influenced by the specific manufacturing process used. Because low-alkali raw materials naturally occur in Western Canada, cements manufactured there generally have lower alkalis than in the East. In areas where moderately low alkali raw materials can be obtained, special measures can sometimes be taken in a cement plant to produce a lower alkali cement than would otherwise result. These all depend on the ability to bypass and waste kiln dust, which would otherwise be recycled.

The provision of a bypass to enable this is costly, but the main added costs are due to loss of the heat energy contained in the flue gasses and the cost of disposal of the dust, which would otherwise form part of the final product.

Although the CSA Standard warns that, where the concrete will be exposed to a continuously moist environment or deicing salts, use of a low-alkali cement may not, in some cases, provide adequate protection against expansion, and also that its use may not prove effective with certain types of aggregate, some specifiers still believe that low-alkali cement is a universal panacea for prevention of alkali-aggregate reaction. While Canadian cement manufacturers provide information on cement alkali levels to purchasers, it should be noted that CSA Standard A5, "Portland Cement", differs from ASTM C150, in that it does not include any optional requirements for a low-alkali cement, having 0.6% or less total alkalis. This is because CSA Committees are aware of the importance of aggregate testing and do not wish to imply that specifying a cement at any given alkali level might be felt to justify the omission of such evaluation.[7] This is particularly important in regions where AAR is known to occur and where proper decisions regarding its control can only be made in the light of test results.

In a paper written in 1975,[8] Bryant Mather, of the U.S. Corps of Engineers, observes:

> With the need on the part of cement manufacturers to comply with regulations regarding air quality, it is now no longer economically feasible to waste kiln dust in order to keep down the alkali content of cement. With a need on the part of cement manufacturers to use more efficient cement-making equipment to reduce the quantity of energy consumed per unit of product produced, the alkali content of cements will increase. It may be expected that cement produced so as not to contain more than 0.6% alkalies calculated as sodium oxide will no longer be available from some producers and will be available only at a premium price from most other producers (see Idorn, 1974).
>
> This alone should justify a re-examination of the justification for requiring low-alkali cement.

While the position taken by the Corps of Engineers is not to require low-alkali cement for all concrete in a large area, such as an entire state, simply to avoid the cost of testing, other agencies have done so. Experience in the U.S.

has proven that once one major specifying agency requires low-alkali cement, others may follow without considering its relevance to their own specific situations. Were this to happen in Canada, a small one-cement-silo concrete producer could be forced to use low-alkali cement on all of his work, to ensure that he could supply concrete to his municipality. Such a scenario would have serious cost repercussions for concrete users and would decrease concrete's competitive position in all markets.

In light of this, it is not surprising that the specification which served to focus the attention of CPCA on the need to try to accelerate the aggregate evaluation process in Canada is that which resulted from the extensive cracking of some 300,000 CNR prestressed concrete railway ties, allegedly due to AAR. These ties were produced between 1974 and 1977 by a precaster in the Montreal area, under a contract worth some $4.5 million. The damages being sought by the CNR for replacement of the defective ties were originally set at $34 million. This case has yet to come to court.

As a result of this experience, both the CNR and CPR instituted a policy of specifying low-alkali cement for use in all concrete construction which will have an outdoor exposure. This specification is now in effect for railways work in all regions of Canada, regardless of whether AAR problems have ever been experienced in that area.

6. CPCA INITIATIVES

Since the promulgation of the railway's specification, CPCA has taken several actions towards attempting to speed up the process of identifying problem aggregate sources and promulgating suggested remedial measures for specific problem areas. These actions are being carried out on a Provincial basis for several reasons. For example:

- The necessary work has already, in large measure, been done in Ontario;
- The magnitude of the problem varies in the extreme from, say, Ontario and Quebec, where problems are numerous, to B.C., where they are very infrequently found at present; and
- The Mineral Development Agreements, from which funding for studies has been available, are between the Federal Government and individual Provinces.

S. Cumming

In broad terms, what we have done is to convene Provincial meetings involving cement and concrete industry representatives, aggregate producers and provincial agencies concerned with concrete construction to:

- Review the AAR situation as presently experienced in structures in the Province;
- Determine the status of AAR testing carried out to date in the Province;
- In Provinces where AAR problems are manifest, attempt to develop a phased plan leading to the identification of sources of problem aggregates;
- Determine appropriate remedial measures to enable use of marginal aggregates; and
- Determine possible sources of funding, including MDA's, for these studies.

Provincial meetings have so far been held in New Brunswick, Quebec, Nova Scotia and Newfoundland. In all cases, useful discussions were held, which led to the conclusion that, as is already being done in Ontario, the provincial Department of Transportation should assume a leading role in the establishment of a provincial program for the evaluation of aggregates from the standpoint of AAR. Further meetings are planned to monitor the progress being made in these efforts which, it is hoped, will eventually result in a system of provincially run evaluation programs in all parts of Canada. These programs should eventually be able to provide specifiers of concrete with current and accurate information on aggregate quality, thus eliminating the need for costly and time-consuming aggregate testing on a job-by-job basis. The availability of such information will also enable the development of proper knowledge-based specifications, rather than documents which prescribe corrective measures, such as the use of low-alkali cement or pozzolans in the concrete, without regard for whether the taking of such measures is necessary or cost effective.

7. CONCLUSION

In conclusion, the cement industry fully supports the position taken by CSA Standards in requiring a proper evaluation of concrete aggregates for AAR. It also supports the efforts which are being made towards the development of

improved and accelerated test methods for such aggregate evaluation, and towards aggregate classification, provincially, in Canada. Where potentially deleterious aggregates are found, selection of the optimum remedial measure to be taken depends on a further evaluation of the relative economics of the possible alternatives. For specific cases, in some regions, specifying the use of low or reduced alkali cement will, of course, continue to represent an optimum solution.

Alkali aggregate reactivity is a potential problem which can be controlled if the proper quality assurance measures set forth in CSA A23.1 are taken. Initiatives which are now well under way, towards the identification of unacceptable aggregates and control of those that are marginal, will result in continued user confidence in concrete as THE most durable construction material available.

REFERENCES

[1] Grattan-Bellew, P.E., Institute for Research in Construction, National Research Council of Canada, (editor); Concrete Alkali Aggregate Reactions; Proceedings of the 7th International Conference, 1986; Ottawa, Canada.

[2] Rogers, C.A.; Alkali Aggregate Reactions, Concrete Aggregate Testing, and Problem Aggregates in Ontario - A Review; Engineering Materials Report EM-31; Ontario Ministry of Transportation, 1987.

[3] CSA Standard A23.1-199Ø, Concrete Materials and Methods of Concrete Construction; Canadian Standards Association; Rexdale, Ontario.

[4] Morgan, D.R. and Empey, D., Hardy BBT Limited; Alkali Aggregate Reactivity in Concrete Structures in Western Canada; Proceedings of Dam Safety Seminar; Edmonton, Alberta; September, 1989.

[5] Soles, J.A. and Malhotra, V.M., CANMET, and Suderman, R.W., Cement Lafarge Ltd.; The Role of Supplementary Cementing Materials in Reducing the Effects of Alkali-Aggregate Reactivity; CANMET Investigations, (Appears in Reference Number 1, Proceedings of the 7th International Conference, 1986.)

[6] Chen, H. and Grattan-Bellew, P.E.; 1989; Effect of Cement Composition on Alkali-Aggregate Reaction; Proceedings of the 8th International Conference on Alkali-Aggregate Reaction; Kyoto, Japan; pp. 635-640.

[7] Grattan-Bellew, P.E.; Preventive Measures to Counteract Expansion of Concrete Containing Alkali-Reactive Aggregates; D.B.R. Paper No. 1132, NRCC 22632, 1982.

[8] Mather, B.; New Concern over Alkali-Aggregate Reaction, NSGA Circular No. 122, NRMCA Publication No. 149, March, 1975.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

TESTING FIELD CONCRETE FOR FURTHER EXPANSION BY ALKALI-AGGREGATE REACTIONS

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ABSTRACT

Expansion tests on cores are now commonly used to assess the potential for further expansion of AAR affected concretes. However, the experimental conditions greatly vary from one study to another (preconditioning, temperature, 100% R.H. or immersion in alkaline solutions, etc.), as well as the interpretation of the results. The various tests and procedures proposed are discussed, and the results of experiments recently performed in our laboratory are also presented. The expansion results must be always interpretated in accordance with the amount of active alkalies still remaining in the concrete under test, and the humidity conditions, as well as the "in situ" stress conditions. The methods suggested to determine the two first parameters are also reviewed and discussed.

1. INTRODUCTION

Three conditions must prevail to promote alkali-aggregate reactions in concrete: 1) presence of **reactive aggregates**, 2) **high alkali concentration** in the pore solution (this promoting a high pH), and 3) **high humidity**. Usually, the alkalies mostly come from the cement, but some may also be supplied by other concrete constituents: chemical additives (Wang & Gillott 1989, Buil & Zelwer 1989), mineral additives, mixing water (salt), and aggregates. Secondary alkalies may be also supplied to concrete by salt solutions (sea water, de-icing salts, industrial activities,...). According to many authors, a relative humidity over 75 to 85% is normally required to sustain AAR in concrete (BCA 1988, Stark & Depuy 1987, Olafsson 1987).

Also, the **wetting/drying cycles** are though to promote the reactions (Semmelink 1981). However, many field observations (Bérubé et al. 1989) suggest that these cycles promote cracking at the surface of concrete, while rather reducing the reactions. Indeed, (1) during the drying periods, the relative humidity decreases in the surface concrete, and (2) during the wetting periods, this concrete may suffer some alkali leaching, as suggested by some laboratory experiments (Kobayashi 1989, Rogers & Hooton 1989). These two conditions tend to reduce the alkali-aggregate reactions in the surface concrete. Then, this concrete will develop patterns of tension cracks as a result of the expansive forces generated by the internal mass of reactive concrete (Kobayashi et al. 1989). Once the concrete is cracked as a result of AAR, the **freezing/thawing cycles** may accelerate the deteriorations (Grattan-Bellew 1981, Rogers 1987, Mukkerjee & Bickley 1987). Moreover, **salt solutions** (de-icing salts, sea water, etc.) supply additional alkalies to concrete, and may contribute to initiate, to accelerate, or to

maintain the reactions. Also, salt solutions can move along cracks and corrode the reinforcements, then causing concrete spalling and rusting.

According to a certain number of more or less characteristic signs (concrete expansion, crack patterns, gel exudations,...), and to supplementary analyses in laboratory (BCA 1988, Palmer 1989, Oberholster 1983), it may be relatively easy to determine if a given structure has suffered from AAR, although it could be not as easy to establish 1) if this reactivity is the only factor responsible for the observed deterioration, and, if not, 2) at what extent it contributed to the overall deterioration. It is still much more difficult to answer these questions from the owners: At which stage are the reactions now? Are they completed? If not, how much additional expansion can be expected, at what rate and for how much time? And what is the future of relatively young structures containing potentially reactive aggregates, but which do not presently show any signs of AAR? Normally, the alkali-aggregate reactions, and the associated expansions and deteriorations will continue in an AAR affected concrete structure as long as the reactive mineral phases are not completely consumed, and if the two other essential conditions are still satisfied: high humidity and high pH (usually over 13) in the pore solution. The latter condition normally holds if the concentration of alkalies stay high in solution.

2. "IN-SITU" MONITORING

Monitoring the current deformations (expansion, relative and absolute movements of concrete members, number, width and depth of cracks,...) remains the best way to estimate the potential for further expansion and deterioration of AAR affected structures (BCA 1988, Hobbs 1988). Indeed, the actual rates of deformation are measured, and can be then extrapolated. Moreover, "in-situ" measurements may lead to more optimistic results than laboratory tests on concrete cores, which are not tested under the same conditions as in the field, particularly concerning the conditions of internal stress (reinforcement, static and dynamic loading, pre-constraints, post-tension, etc.). This is particularly true when investigating the mechanical performance of concrete affected by AAR (Bérubé et al. 1989). Various methods can be used for monitoring concrete expansion (Solymar 1989). In Canada, a certain number of AAR affected structures, mainly dams, are now instrumented (Charlwood & Solymar 1989, Wroblewicz et al. 1988, Bouchard 1987, etc.).

The deformations generated by the alkali-aggregate reactions must be distinguished from the natural deformations due to variations in the exposure conditions (thermal and climatic effects, humidity variations, changes in loading or trafficability, etc.). Then, it takes usually many years of "in situ" monitoring to get relevant information for prognosis (BCA 1988). On the other hand, laboratory expansion tests on concrete cores, which are performed under well-controlled conditions, can supply results in a relatively short period of time, while being less expensive. This explains why these tests are now commonly used to assess the future of field concrete affected by AAR.

3. EXPANSION TESTS ON CORES

3.1 General Considerations

Specimen sizes. According to the usual practice, the diameter of the cores should be at least three times the maximum size of the aggregates, and their length should be at least two times the diameter (BCA 1988). Cores of 100 mm dia. (aggregates <33 mm) seem more practical (BCA 1988), but smaller or larger diameters (75 to 150 mm) are also acceptable, provided the above conditions are satisfied. **Surface effects.** It is recommended to remove

the exposed part of the concrete, since this concrete is usually more cracked and may have suffered some alkali leaching. **Concrete heterogeneity and number of cores.** Also, the cores taken from a given concrete member may present large variations in term of composition, internal stress, humidity conditions, etc., thus leading to variations in expansion. It is important to test many samples corresponding to various members from the concrete structure under investigation (Wood et al. 1987, BCA 1988). **Sample heterogeneity and number of gauge reference studs per sample.** Wood et al. (1987) reported large variations of expansion within a single core, for instance 0.12% to 0.31% (at 140 days) for a given core tested at 100% R.H. and 38°C (6 readings along the core). It is recommended to fit at least 9 Demec gauge reference studs to each core along three equally spaced lines with two 50 mm gauge lengths each (6 readings) (BCA 1988). The studs should be placed at least at 20 mm from the ends of cores (50 mm from the end corresponding to the exposed concrete surface) (BCA 1988). An alternative way to take account for the expansion heterogeneity inside the cores may consist to install pin gauges at both ends, then allowing measurement of the average longitudinal expansion (Bérubé et al. 1988).

Volumetric changes due to new conditions and rewetting. Normally, a preliminary expansion phase is observed due to various mechanisms not related to secondary AAR (Hobbs 1988, Stark & Depuy 1987, Oberholster 1983): 1) Thermal contraction or expansion. 2) Stress release (structural efforts, static and dynamic loading, pre-constraint, post-tension, reinforcement,...). 3) Humidity uptake when the humidity conditions were lower in the concrete structure, or if the samples have been allowed to dry since coring. Indeed, the expansion tests on cores are always performed under very high humidity conditions (100% R.H. or immersion in water or in alkaline solutions). 4) Additional water sorption (and consequent secondary microcracking and expansion) by siliceous gels already present in the samples (as a result of higher humidity and/or lower stress in comparison with the field conditions). These points must be taken into account when interpretating the results of the expansion tests. **Sample storage before tests.** In order to minimize drying of the core samples before the tests, then the expansion due to rewetting, it is firmly recommended to seal the samples immediately after coring by wrapping them with cling film such as "Saran Wrap" or "Stretch'n Seal", and storing in sealed polyethylen bags (Hobbs 1988, BCA 1988) **Preliminary mechanical adjustment.** Moreover, the samples should not be tested before they release stresses that they were supporting in the field. As soon as possible after coring, Hobbs (1988) suggested fixing Demec gauges to the core samples, before sealing them. In the laboratory, the samples are measured periodically at room temperature until equilibrium is reached, while kept sealed between the measurements. Then the expansion tests can be started. **Length and weight measurements.** In order to recognize more easily the short term volumetric changes not related to secondary AAR , it is highly recommended to periodically measure the weight of the samples in addition to their length (Palmer 1989, Wood et al. 1989, BCA 1988).

3.2 Literature Review

3.2.1 Experimental Conditions

Tests at 100% R.H. In most cases, the core samples are stored at 38°C (or 40°C) (Oberholster 1983, Wood et al. 1987, Imai et al. 1987, Wakizaka et al. 1987, BCA 1988, Palmer 1989, Tomita et al. 1989: also at 18°C, Nishibayashi 1989, Okada et al. 1989, Mukkerjee & Bickley 1987: cut prisms from pre-cast cladding panels, Kawakami 1989: also at 18°C), and it is recommended to carry out the tests over at least a 1 year period (BCA 1988). Some authors suggest to work at lower temperatures and to go further in time (Palmer 1989: 20°C, Wood et al. 1989: 5, 13, 20°C and field temperature). In the study by Ichihara et al. (1989), the cores are first tested at 20°C for 3 months, before to be transferred to 40°C for 15 months. Cavalcanti & Silveira (1989) tested cores at 30 and 60°C. In a first step, Mizumoto et al. (1987) stored the cores at 100% R.H. and 20°C until convergence, then at 40°C to determine the residual expansion.

Tests in water and alkaline solution. In other studies, samples are immersed in alkaline solutions of KOH 1N (Visvesvaraya et al. 1987: 60°C and room temperature) or NaOH 1N (Albert & Raphael 1987: 23°C, Stark & Depuy 1987: 38°C, Hobbs 1988: 20°C), with companion cores or half-cores (cut lengthways: Hobbs 1988) tested in water. The two results are compared. Again, temperatures and test durations vary from one study to another. In Quebec, a popular test consists of immersing companion cores in water and in NaOH 1N at 80°C for at least 24 days (tests performed at Ecole Polytechnique, at Laval University and in a few private laboratories). These conditions are similar to those for the NBRI accelerated mortar bar test (Oberholster & Davies 1986). Some results are presented hereafter (see section 6).

Except in one case (Wood et al. 1987), the higher the temperature, the higher the rates of expansion, although the long term absolute expansion is not always greater.

3.2.2 Interpretation of Tests in Alkaline Solution and Water

After the (previously sealed) cores reached length equilibrium at room temperature, Hobbs (1988) performs two separate tests at 20°C, in water and in NaOH 1N, on half-cores (cut lengthways). The critical expansion limit for both tests is 0.03% at 28 days. According to the author, (1) expansions <0.03% for both tests (water and NaOH) should mean that the reactions are completed (no more reactive phases). (2) Expansions >0.03% in NaOH and <0.03% in water lead to various possibilities: a) the reactions were not completed, and there is a potential for further expansion in the concrete structure (still reactive phases and enough alkalies); b) reactive phases are still present in the concrete, but the reactions were dormant because of insufficient alkalies in the pore solution, and alkali addition through NaOH 1N restarted (or initiated) the reactions. c) Gels already present in the concrete absorbed more water (than in the field), and produced secondary expansion. For instance, this could take place if the concrete was restrained in the field or protected against rain and condensation. (3) If expansions exceed 0.03% in water and in NaOH, the above possibilities a) and c) remain.

In the study by Stark & Depuy (1987), tests are performed at 38°C on companion cores immersed in water and in NaOH 1N. In this study, expansion due to rewetting usually varies from 0.01 to 0.03%, with maximum values of about 0.1%. According to the authors, after weight equilibrium is reached, (1) significant expansion in water means a) that further reactions could take place in the field concrete (still reactive phases and enough alkalies) and/or b) that preexisting gel reaction products uptook water. The time at which the expansions occur may help to determine which mechanism is the most important. (2) On the other hand, lack of significant expansion in water suggests that alkalies and/or reactive silica are insufficient to produce further expansion in the corresponding concrete member. (3) Expansions in NaOH that are significantly greater (by 0.02% or more, at 8 months) than those in water indicate that reactive phases are still present in concrete, but that alkalies are not sufficient to produce AAR. (4) Finally, expansions in NaOH that are not significantly greater than the small expansions in water suggest that reactive phases are no longer present in the concrete.

As discussed hereafter, we do not totally agree with the above interpretations concerning the tests in water, which may lead to alkali dilution and to minor secondary AAR, at least for normal concretes. For instance, Albert & Raphael (1987) tested companion cores at 23°C in water and in NaOH 1N, and the difference between the two expansion curves was taken as the potential for further expansion. In our mind, the very low values they obtained in water ($\leq$ 0.006% at 1 year) are mostly attributed to rewetting and not to secondary AAR, while much higher expansions ($\geq$ 0.03% at 1 year) have been obtained in NaOH 1N.

3.2.3 Interpretation of Tests at 100% R.H.

Expansion due to rewetting. To take account for expansion due to rewetting, various methods are proposed. Mukkerjee & Bickley (1987) subtract from the expansions at 100% R.H. and 38°C the value (about 0.04%) obtained in water at 23°C for companion cores which had been first dried at 75°C for 3 days and cooled at 23°C. In the study by Wakizaka et al. (1989), the initial reading is taken after 24 hours of immersion in water at 20°C, before the cores are tested at 100% R.H. and 38°C for 6 months. Kawakami (1989) takes the initial length after 2 weeks at saturated conditions, where the expansion by saturation of the specimens becomes stable. In the study by Wood et al. (1989), weight changes were monitored, and the "starting point of the moisture uptake curve was established relative to the maximum water content from vacuum saturated weight and oven dry weight, determined using ends cut from the cores".

Expansion limits. In most papers, expansion limits are not specified. Nishibayashi (1989) states that absolute expansions of cores (at 40°C and 100% R.H.) taken from concrete structures with significant cracking usually exceed 0.05%, without any specification concerning expansion due to rewetting, and time limits. According to Wood et al. (1989), expansions <0.05% for tests performed at 100% R.H. (1 year at 38°C, 2 years at lower temperatures) should be also considered as acceptable.

According to Oberholster (1983), the initial expansion is attributed to water uptake 1) by the cement paste and the aggregates, and 2) by the AAR products (gels) already present in the concrete. The first contribution is a function of the overall quality of the concrete (porosity, microcracking,...). If the concrete is not completely dried, the corresponding expansion will be inferior to drying shrinkage and expansion on wetting. Moreover, the normal wetting expansion should be reversible and approximately equal to drying shrinkage (Oberholster 1983).

3.3 Recent Work at Laval University

Experimental. Concrete prisms have been prepared in accordance with the CSA CAN3-A23.2-14A Concrete Prism Test with two very reactive aggregates from Quebec (siliceous limestone and rhyolitic tuff), using a normal cement containing 0.85% alkali (Na_2O equiv.), with NaOH addition to the mix water to increase the alkali content to 1.25% (per unit mass of cement) (overall concrete alkali content: 3.9 kg/m^3). With the limestone, prisms have been also prepared with a low alkali cement (0.52%), without any NaOH addition (overall concrete alkali content: 1.6 kg/m^3). The water/cement ratio was 0.55 for all the specimens. The results of expansion tests performed at 38 and 80°C, for various storage conditions (100% R.H., water, NaOH 1N and NaCl 1M) appear on Fig. 1. Also, samples made with a non reactive aggregate (phonolite: expansion <0.01% at the CSA Concrete Prism Test) have been tested in NaOH 1N at 80°C.

Expansion tests at 38°C. (1) At least until 6 months, immersion in NaOH 1N gave higher expansion than in NaCl 1M, even if the two solutions are alkali equivalent (Figs. 1A and 1B). This is likely associated to the delay before the Cl^- ions are replaced in the pore solution by OH^- ions (from portlandite) (Yonezawa et al. 1989). In fact, the expansions in NaCl are not significantly different from those at 100% R.H. (2) Immersion in water gives relatively low expansions (<0.04% at 10 months). This is attributed to alkali dilution. (3) However, about 1 month after transfer to NaOH (at 9 months), the sample initially immersed in water started to expand at about the same rate as its companion sample initially immersed in NaOH (Fig. 1A). (4) A low alkali content in the original concrete retards the expansions, as observed on Fig. 1A. (5) The results are very similar for the two reactive aggregates tested.

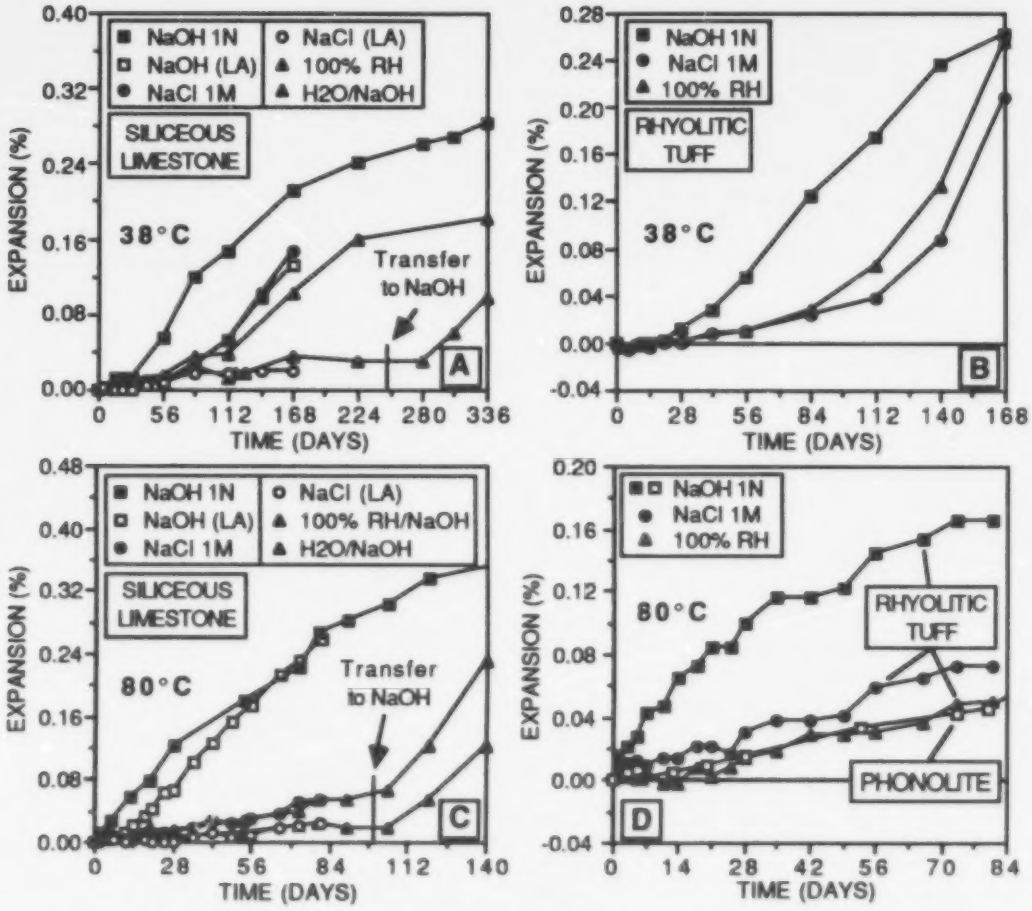

Fig. 1 Expansion tests on laboratory concrete prisms made with two very reactive aggregates (siliceous limestone and rhyolitic tuff), and one non reactive aggregate (phonolite), and stored under various conditions (38 and 80°C; NaOH 1N, NaCl 1M, 100% RH, and water). A low alkali cement (LA) has been used for some tests, without NaOH addition to the mix water.

Expansion tests at 80°C. The results are quite similar at higher temperature, except for the following remarks (Figs. 1C and 1D): (1) In NaOH, the expansion rates are much greater at 80°C than at 38°C, about 7 times higher at the beginning of the tests, for both aggregates. However, at 100% R.H. and even in NaCl 1M, the expansion rates are not much higher than at 38°C. (2) Significant expansion can be obtained at long term with non reactive aggregates (Fig. 1D: phonolite). When testing aggregates for potential of AAR (no rewetting effect), the suggested limit for the accelerated concrete expansion test in NaOH 1N at 80°C is 0.04% at 24 days (Blanchette 1989, Bérubé & Fournier 1989).

3.4 Discussion

Permeability effects. For concretes with normal permeability and containing reactive aggregates, it is well known that immersion in NaOH 1N tends to accelarate the reactions by increasing the concentration of alkalies in the pore solution (Fournier & Bérubé

1989, Bérubé & Fournier 1989, Blanchette 1989). This suggests quite easy alkali exchange between the immersion solution and the pore solution. However, the reverse effect (alkali dilution) is necessarily also present if the concrete is immersed in pure water. This point is not discussed in the literature, but is clearly evidenced on Figs. 1A and 1C (see also Fig. 4B). As a result, AAR and related expansions are reduced, if not completely stopped. Stark & Depuy (1987) never observed expansions in water that could be attributed to secondary reactions, (likely because of alkali dilution), and then concluded that no further reactions should take place in the five dams under study (provided that no additional alkalies are supplied to concrete). In the study by Albert & Raphael (1987), as mentioned before, the difference between the two expansion curves in NaOH 1N and in water (at 23°C) is taken as the potential for further expansion; even if not mentioned by the authors, this suggests that alkali dilution takes place in water, and that the (very low) expansions in water (≤ 0.006% at 1 year) are mostly associated to rewetting. Such a procedure seems acceptable for normal and mass concretes (with normal to high permeability). It is however possible that ion exchange will be slow between the immersion solutions and concretes with low permeability. In such a case, the nature of the immersion solution (water or alkaline) should have no significant effect on the results, with high expansions also obtained in water, provided that the concrete contains reactive aggregates and alkalies in sufficient amounts to sustain AAR. This point is confirmed by Al-Asali & Malhotra (1989) for concretes made with a low w/c ratio (0.30), and immersed in various solutions at 38°C (NaOH 1N, KOH 1N, NaCl 5%, and water).

Tests in NaOH and in water. Immersion tests of concrete cores in NaOH 1N are useful to evaluate if potentially reactive phases are still present in the concrete under test, but do not necessarily express the past or the future behavior of this concrete in the field structure (BCA 1988). Indeed, the immersion alkaline solutions used for the tests are highly concentrated, and supply large amounts of alkalies to the concrete pore solution, while the experimental humidity conditions are extreme. Despite the presence of very reactive aggregates, no further reaction or expansion due to AAR will take place in a field concrete if the other essential conditions (alkalies, humidity) were never satisfied or if they are no longer satisfied. At least, the results of expansion tests on cores should be interpretated in accordance with the actual alkali content of the field concrete. Moreover, some aggregates, for instance dolostones and dolomitic limestones from the Beekmantown and the Chazy Groups from the St.Lawrence Lowlands, in Quebec, which appear innocuous in field concretes as well as at the CSA CAN3-A23.2-14A Concrete Prism Test, are very expansive in NaOH 1N (Fournier & Bérubé 1989, Bérubé & Fournier 1989, Blanchette 1989). On the other hand, immersion in water should lead to alkali dilution for concretes with normal to high permeability. Thus, only low expansions should be obtained (at least if the samples were not allowed to dry), and the results should not be used to assess the potential for further expansion in the field concrete. At the most, they can be used to estimate the expansion due to rewetting.

Tests at 100% RH. The more realistic test is likely the one at 100% R.H., even if the humidity conditions are also extreme. In order to accelerate the reactions, a temperature of 38°C appears acceptable. Besides, these conditions are the most popular. The above results on laboratory samples suggest that temperatures higher than 38°C do not greatly accelerate the expansions (Fig. 1). Moreover, time is not as critical when testing field concrete as when testing aggregates for a new job. On the other hand, a lower temperature of 20°C or close to field conditions should be more realistic (Palmer 1989, Wood et al. 1989), but corresponding data are limited (Palmer 1989), while a longer period of time should be required to reach a conclusion. The initial water uptake can be estimated by looking at the expansion curves (inflexion point between the initial expansion due to rewetting, and the secondary expansion due to secondary AAR) (see Fig. 4). The suggested expansion limit could be around 0.04% at 1 year (after correction for the rewetting expansion). Optionally, tests in water and in NaOH 1N (also at 38°C) could be undertaken to better assess the initial rewetting expansion (likely more rapid in water than at 100% R.H.), and to evaluate the absolute reactivity potential of the aggregates

present in the concrete under study (tests in NaOH, i.e. under extreme conditions of alkalies and humidity).

Concretes that have already suffered from AAR should present little or no further reactions and expansion when tested on cores, if the alkalies or the reactive phases have been mostly depleted by the reactions. Conversely, as pointed by Oberholster (1983), secondary AAR reactions could take place in cores without any detected expansion, if the secondary reaction products are produced in insufficient amounts to fill the cracks that are already present within the aggregate particles and the cement paste. The author also concludes that the expansion from rewetting is sometimes difficult to distinguish from expansion due to secondary AAR, thus complicating the interpretation of the results. These remarks apply to tests at 100% RH as well as in alkaline solutions.

3.5 Prognosis

The assessment of the further expansion of field concrete affected by AAR through laboratory tests must be based on the three following parameters: (1) the absolute residual reactivity potential of the aggregates present in the concrete, (2) the residual alkali concentration in the pore solution of the field concrete, and (3) the humidity conditions prevailing in this concrete. The two last points are discussed hereafter at sections 4 and 5, respectively. The expansion results should be also interpretated in accordance with many other parameters, such as the concrete mix proportions, the age of the structure, the current degree of deterioration (expansion, cracking,...), the progress of this deterioration since construction (expansion and cracking rates), the results from "in situ" measurements and monitoring, the "in situ" stress conditions, the past and the future exposure conditions (mechanical solicitations, de-icing salts, etc.), and the behavior of other structures built with similar aggregates (BCA 1988). It is particularly important to take account for the restraint forces applied in the field to the concrete under test (Palmer 1989). Restrained expansion tests on cores have been performed by a few workers (Wood et al. 1987, 1989). In this respect, the study by Amasaki & Takagi (1989) suggests that the expansion of concrete cores taken from a reinforced concrete member depends upon the amount of reinforcement: the higher the reinforcement in the field, the higher the expansion of the corresponding cores in laboratory. This is likely attributed to stress release.

Other parameters related to the mix design may also affect the reactions or the expansions of field or laboratory concrete: w/c ratio, air entrainment, size of aggregates, etc. In addition, the size of the specimens tested (core diameter) may also play a role. In general, the coarser the specimens tested, the higher the expansions obtained (Bérubé et al. 1989). This volume effect could be important in mass concrete (dams,...), which is sometimes deteriorated by AAR, despite being of relatively low alkali contents (Charlwood & Solymar 1989).

4. HUMIDITY MEASUREMENT

The humidity conditions prevailing in field concrete can be determined by using the method proposed by Monfore (1963), which has been successfully used for dams, on cores (Stark & Depuy 1987) and on powdered drilling samples (Albert & Raphael 1987, Stark 1984). However, according to Wood (1985), for a substantial part of the year, the British climate is such that most structures with outdoor concrete members have relative humidities in the 80 to 90% range, which is sufficient for AAR take place. This also applies to the Canadian climate. Moreover, in the case of dams, Albert & Raphael (1987), Stark (1984), and Stark & Depuy (1987) observed that the relative humidity in concrete was reaching 85% at depths greater than 5 to 40 centimeters (depending upon the concrete permeability, and on the environmental conditions, in particular temperature and humidity).

5. ALKALI MEASUREMENT

AAR tends to deplete with time the amount of alkalies in the pore solution, then the pH (Seno & Kobayashi 1989), as a result of entrapping in the reaction products. Then the knowledge of the residual amount of alkalies in solution is a very interesting parameter for assessing the potential for further reaction in a AAR affected concrete, particularly if the original amount is known.

5.1 Acid Soluble Alkali Content

The alkali content of service concrete is often determined using an acid extraction technique (Hobbs 1988, BCA 1988, Nixon & Gillson 1987, St-John 1989, Rogers & Hooton 1989). In such conditions, some aggregates may release alkalies. St-John (1989) states that errors up to 700% may arise with classical methods using dilute hydrochloric acid, particularly with mafic aggregates like basalts. The author also points out that extraction with picric acid in methanol/water mixtures gives better results, although if some alkalies are still extracted from basaltic aggregates. In some cases, a correction can be applied to take account for the amount of alkalies released by the aggregates (Nixon & Gillson 1987), or the analysis can be made on "fines-rich fraction obtained by screening out as much as possible of the aggregate prior to analysing the remainder. Recent work has suggested, however, that some porous aggregate may absorb some of the available alkali from the mix, so using this technique may underestimate the available alkali content." (BCA 1988). In fact, the values obtained through acid leaching summarizes various contributions: 1) the "active" alkalies that were already dissolved in the concrete pore solution, 2) and some of the "non active" alkalies entrapped in the cement paste hydrates, in the AAR reaction products, and in the aggregates.

5.2 Water Soluble Alkali Content

5.2.1 Experimental Methods

For prognosis, the amount of residual "active" alkalies in the pore solution is the most interesting parameter. To better estimate these, extraction methods in hot (St-John 1989, Rogers & Hooton 1989, Mizumoto et al. 1987) or cold water (Okada et al. 1989) became more and more popular. St-John (1989) states that hot water extraction on <300 µm material releases about 85 to 90% of the alkalies, but did not mention if this amount is relative to total or to "active" alkalies. Also, the author points out that the extraction efficiency depends on the cement used.

For a few years, the Ministry of Transportation of Ontario currently has used the following hot water extraction method to determine the water-soluble alkalies (Rogers & Hooton 1989):

1. A concrete sample (of at least 2 kg) is reduced to pass the 200 mesh sieve (<74 µm).
2. A 10 g sub-sample is immersed in 100 ml of distilled water, boiled for 10 minutes, and allowed to stand overnight at room temperature.
3. The suspension is filtered, and the solution is completed to 100 ml by adding distilled water.
4. Sodium and potassium are determined by flame photometry, and expressed in kg of alkali (Na_2O or K_2O or Na_2O equiv.) per m^3 of concrete, provided the concrete density is known.

5.2.2 Discussion

Precautions should be taken to prevent ionic interferences during the chemical analyses (BCA 1988). For many reasons, the amount of water soluble alkalies usually departs from the total alkali content supplied by the cement (cement content x alkali content of the cement):

Negative contributions:

1) Some alkalies from the cement are not available to the pore concrete solution ("non active" alkalies), particularly those in silicates and aluminates, and remain entrapped in the cement paste hydrates (Buil & Zelwer 1989).
2) Some alkalies released early into the pore solution ("active" alkalies), are progressively entrapped in AAR reaction products (increasing effect with time).
3) Some "active" alkalies are progressively leached from the specimens (in field concretes, since construction, or in laboratory concretes, with time).

Positive contributions:

4) Aggregates (since construction as well as during the analysis) (increasing effect with time ?).
5) Other concrete constituents: water, chemical, and mineral additives.
6) External sources (sea water, de-icing salts, etc.).

Possibility 1) should always apply, according to the work by Oberholster (1983). This author used the ASTM C311 Standard (but with no $Ca(OH)_2$ addition) to determine the amount of "active" alkalies in 21 different cements (Fig. 2). This amount varied from about 65% to almost 100% of the total alkali content. Moreover, this proportion tends to increase with high alkali contents. It is likely that this procedure gives the best measure of the active alkali content of cements (e.g. available to the pore solution). Indeed, the expansions of ASTM C227 mortar bars made with the 21 cements tested were much better correlated with the amount of active alkali content of the cement (correlation coefficient of 0.96) than with the total alkali content (CC = 0.87), or with the water soluble alkali content (CC = 0.82). Point 2) is well experienced (Bérubé & Fournier 1986, Oberholster 1983). However, it is not known if hot water can leach some of the alkalies entrapped in the reaction products. Point 3) has been well established on laboratory concretes (Kobayashi 1989, Rogers & Hooton 1989), while 4) has been previously observed by many authors (Hobbs 1988, Kawamura et al. 1989, Mizumoto et al 1987).

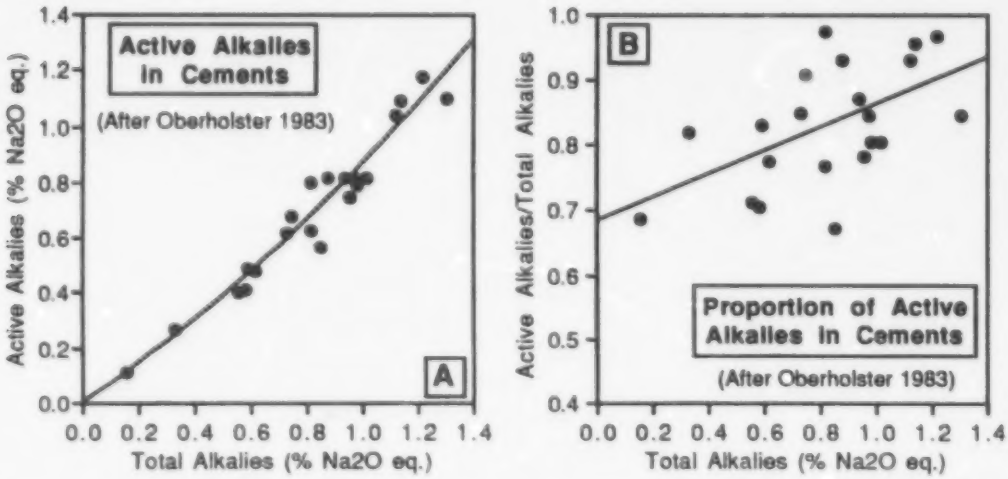

Fig. 2 The amount of active alkalies in cements from South Africa, determined through the ASTM C311 standard procedure (however without $Ca(OH)_2$ addition), normally exceeds 70% of the total alkali content. (After Oberholster 1983).

5.2.3 Experiments by the Ministry of Transportation of Quebec

Tests on aggregates. The above hot water extraction technique has been applied to a few aggregates (taken alone) by the Ministry of Transportation of Quebec (MTQ). The results suggest that the most common coarse and fine concrete aggregates used in Quebec (limestones and granitic sands) supply significant amounts of alkalies through the above hot extraction method. Indeed, reactive limestones from Trois-Rivières released 0.023% by weight of Na_2O equiv. (0.014% of Na_2O, and 0.014% of K_2O), while the granitic sands tested released 0.033% by weight of Na_2O equiv. (0.02% of Na_2O, and 0.02% of K_2O). For normal concretes, this would contribute more than 0.5 kg/m^3 to the result. However, this value is largely underestimated since the pH of the hot extraction solution remains around 7.0 when aggregates are tested alone, while the pH naturally raises to around 12.5 when concrete specimens are tested (saturation with respect to portlandite). Moreover, the pH of the concrete pore solution can be sometimes 10 to 20 times still more aggressive in the field concrete (pH over 13.5), according to the alkali content, and the aggregates could have released with time (since construction) significant amounts of alkalies to the pore solution.

Tests on young laboratory concretes. Despite this, results from other experiments performed by the MTQ on 28 days old laboratory concretes made with two coarse aggregates (a very reactive limestone from the St.Lawrence Lowlands, and a mixture of schists and sandstones from the Appalachians), were in very good agreement with the total Na_2O, K_2O, and Na_2O eq. contents supplied by the cement and the NaOH added to the mix water to increase the alkali content to 1.25% per unit mass of cement (Table 1). For these young laboratory concretes, among the 6 possible (positive or negative) contributions mentioned above, nos. 2 (AAR just starting at 28 days), 3 (too short period of time for significant leaching), 5 (no chemical or mineral additives), and 6 (no external alkalies), are excluded. Then, the good agreement observed between the total and the water-soluble alkali contents could be explained by the fact that the amount of alkalies released by the aggregates (resulting from the hot water attack, or possibly from natural leaching in concrete, during the 28 days before), is about equal to the amount of "non active" alkalies.

Table 1. Determination of the water soluble alkali contents of 28 days old laboratory concretes, using the hot water extraction method. (D. Vézina, MTQ, pers. comm.).

Coarse Aggregate	Cement Content (kg/m3)	Concrete Density (kg/m3)	Total Alkalies (kg/m3)*			Soluble Alkalies (kg/m3)		
			Na2O	K2O	Na2O eq.	Na2O	K2O	Na2O eq.
Limestone	310	2387	1.65	3.38	3.88	1.86	3.29	4.02
Schist	310	2416	2.03	2.81	3.88	2.05	3.04	4.06

* NaOH added to the mix water to increase the alkali content to 1.25% per unit mass of cement.

Tests on aged laboratory concretes. Two year old laboratory concretes made with thirteen different limestone aggregates from the St. Lawrence Lowlands (reactive and non reactive), have been also tested for water soluble alkali by the MTQ. For each aggregate, three different mixes had been made using always the same cement (averaging 1.02% Na_2O equiv.: 0.26% Na_2O and 1.15% K_2O), but with three different contents (310, 420 and 524 kg/m^3), in order to obtain total alkali contents of 3, 4 and 5 kg/m^3 of concrete. Moreover, the slump was controlled between 70 and 90 mm by adjusting water, thus leading to water/cement ratios of about 0.52, 0.41 and 0.35, respectively. The results are shown on Fig. 3.

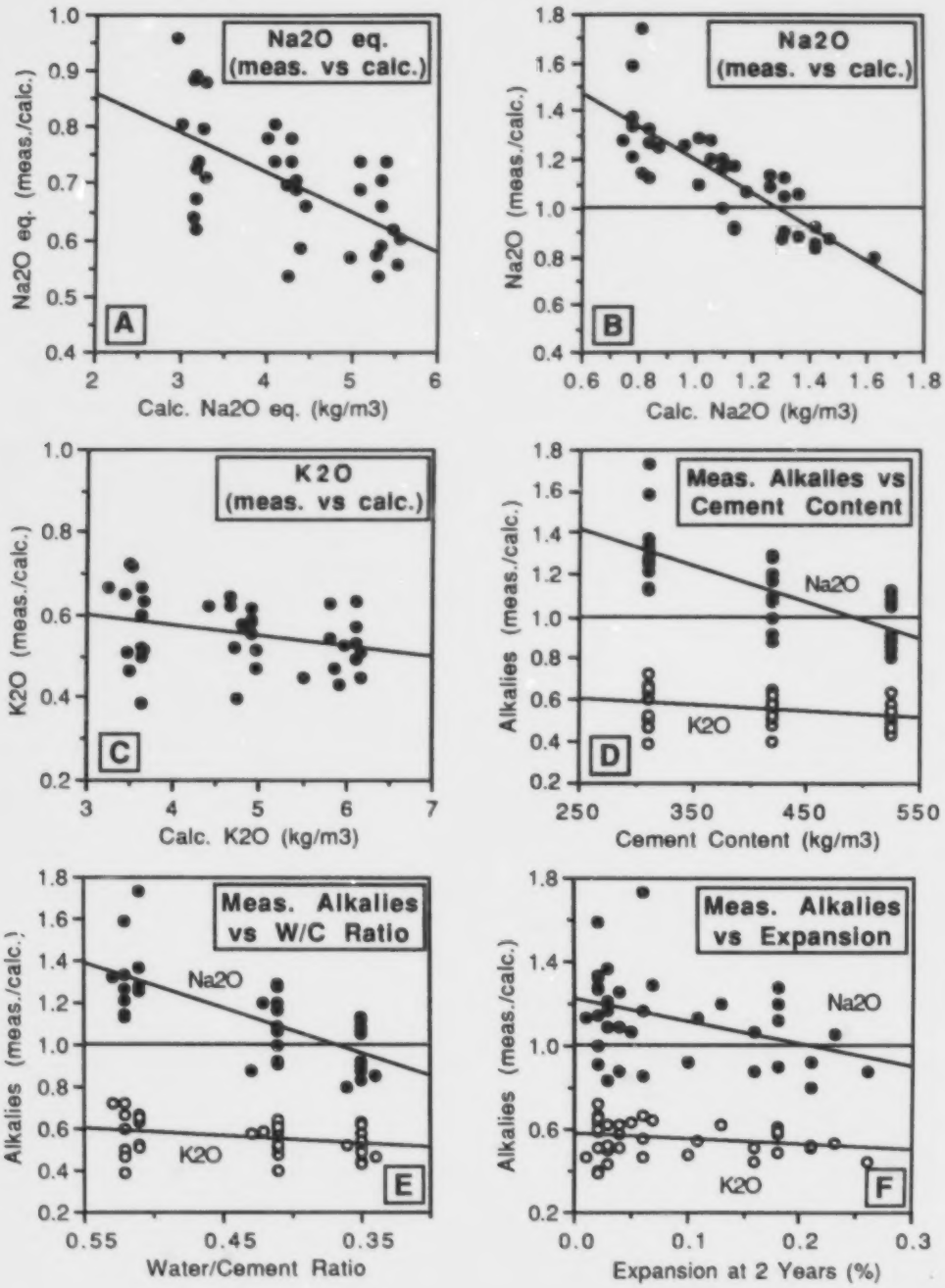

Fig. 3 Determination of the water soluble alkali contents of laboratory concrete prisms made with reactive and non reactive limestone aggregates, after 2 years at 38°C and 100% RH, using a hot water extraction procedure. For all mixes, the slump was controlled by adjusting water, then the w/c ratio varied with the cement content. (Calc. = total alkali contribution by the cement = cement content x total alkali content of the cement). (Results supplied by D. Vézina, MTQ).

Now, the water-soluble K_2O content represents 40 to 70% of the total K_2O supplied by the cement (Fig. 3C), without any significant correlation with the total K_2O content (Fig. 3C), the cement content (Fig. 3D), the water/cement ratio (Fig. 3E), or the concrete expansion after 2 years at 38°C and 100% RH (Fig. 3F). Indeed, linear correlation plots are almost horizontal on each figure. Despite of likely high amounts of alkalies released by the aggregates (particularly after 2 years at high temperature and humidity), a large proportion of water soluble K_2O is then missing compared to very young concretes made with similar aggregates and cements (Table 1). Only time dependent effects can be suggested to explain this. In fact, the difference is mainly attributed to intensive alkali leaching from the concrete prisms with time, due to the type of containers used (plastic pails with cloth wicks). Indeed, under quite similar conditions (38°C over water in sealed box), Rogers & Hooton (1989) report that 42% of the water soluble alkalies (Na_2O eq.) present in concrete prisms made with a reactive alkali-carbonate aggregate was leached from the samples after 2.5 years. On the other hand, if some alkalies in solution are progressively fixed in the AAR reaction products, (as well demonstrated), the results for K_2O on Fig. 3F suggest that this effect is negligible, (even for expansion in excess of 0.2%), or that these alkalies are mostly leached through hot water extraction. In the latter case, this procedure could not be appropriate to estimate the residual amount of alkalies that have been effectively removed out of the pore solution by the AAR reaction products!

The results for Na_2O are quite different. Indeed, the water-soluble Na_2O content is on an average higher than the total Na_2O supplied by the cement (Fig. 3B), with values from 80 to 140%. This suggests that, on an average, more sodium was released from the aggregates than leached out from the specimens. It must be pointed out that the cement used contained much less Na_2O than K_2O, while the aggregates are thought to contribute as much sodium as potassium to the concrete pore solution (e.g. analyses on aggregates taken alone). Consequently, the sodium contribution by the aggregates is significantly more important than the potassium contribution, relatively to the corresponding amounts supplied by the cement. However, this relative contribution by aggregates to total sodium becomes less and less important when the cement content is increased (according to a lower mass ratio between the aggregates and the cement), and this explains why the higher the cement content, the lower the proportion between the water soluble and the total Na_2O content (Fig. 3B). The similar trends observed for sodium on Figs. 3D, 3E and 3F are similarly explained, according to the fact that, in this study, the total alkali content (from the cement), the cement content, and the w/c ratio are totally dependent variables. Globally, the proportion between water soluble and total Na_2O eq. presents an intermediate behavior between Na_2O and K_2O (Fig. 3A), with values never exceeding unity (because of smaller contents in sodium with respect to potassium, in the cement).

5.3 Alkali Concentration in the Pore Concrete Solution

In the best case, a water extraction method gives the amount of alkalies in solution on a kg/m^3 basis. According to Diamond (1989), for a near saturated normal concrete made with a w/c ratio of 0.5, the concentration of the OH^- ions in the pore solution (or of the alkali ions) should be approximately equal to 0.7 moles/litre per percent Na_2O equiv. in the cement, irrespectively to the amount of soluble alkalies in the cement. Indeed, Diamond (1989) tested 8 different cements selected to cover the widest possible range of alkali types and locations in cement (soluble or insoluble). The above factor is lower or higher than 0.7 for higher or lower w/c ratios, respectively, a correction being possible in these cases. Thus, in as much as this ratio is known as well as the total alkali content of the cement used, the equilibrium concentration of the concrete pore solution at relatively low ages could be calculated. However, it is not possible to determine this way the amount of alkalies which have been removed out of the pore solution, as a result of AAR. The only way to get this information is to extract samples of pore solution by applying high pressure on concrete samples. Such a procedure has been already

applied to cement pastes and mortars (Longuet et al. 1973, Barneyback & Diamond 1981, Buil & Zelwer 1989, Seno & Kobayashi 1989), but, to our knowledge, has not been used on aged concretes. Moreover, some people question the fact that the pore solution is extracted at very high compressive strengths, this possibly affecting the chemistry of the solution (Buil & Zelwer 1989).

6. EXAMPLE

Description of samples. Two sets of cores (A1-A2, B1-B2) of 100 mm diameter and 300 mm long were taken inside a fifteen year old building from two concrete components A and B in which alkali-silica reactions were suspected (Bérubé et al. 1988). Both components contain coarse limestone aggregates in the range of 5 to 20 mm, in normal proportions. Petrographic examination of polished and thin sections of concrete revealed: (1) that samples A were more microcracked (82% of the coarse aggegate particles were microcracked) than samples B (9% only), (2) that they contained a larger amount of fine-grained limestone particles (with higher amount of insoluble siliceous residues) (80% of the coarse particles vs 55% in B samples), and (3) that they were showing more characteristic signs of AAR (reaction "rims", gels and microcrystalline reaction products). In accordance with the cements used, the cement contents, and the alkali contents of these cements (Table 2), these two concretes contained less than 3 kg/m^3 of total alkalies (Na_2O equiv.) (Table 2). Concrete A, more affected by AAR, contains more alkalies than concrete B (2.9 kg/m^3 vs 1.8), while corresponding to a significantly lower water/cement ratio (0.41 vs 0.65 for B). The determination of the water soluble alkali contents is in progress.

Table 2. Characteristics of the core samples tested.

Sample	Cement Content (kg/m3)	Alkalies in Cement (%Na2O eq.)	Total Alkalies (kg/m3)	Coarse Aggregate Particles (%)	
				With Microcrack	Fine-Grained Limestone
A1-A2	430	0.67	2.9	82	80
B1-B2	250	0.73	1.8	9	55

Expansion tests and results. Pin gauges have been fixed at both ends of the cores for length measurements. Since the samples had been kept many weeks at room conditions since coring, all the cores were first immersed in water at 23°C (saturated with lime). As expected, a significant expansion has been readily observed during the first day (Fig. 4A), due to humidity uptake, and/or water absorption by the gels already present in the concrete. The initial expansion of concrete A, more affected by AAR, was higher than for concrete B (0.061% vs 0.030% after 3 days), despite the fact that this concrete was less permeable (w/c ratio of 0.41 vs 0.65). This is likely attributed to a larger amount of microcracks and siliceous gels in this concrete. After three days in water at 23°C, the samples reached a relative equilibrium. One core of each concrete was then immersed in NaOH 1N at 80°C, with the other companion core in pure water at 80°C. The zero reading was taken the day after. After 73 days in water, the sample A2 was transferred to NaOH 1N (Fig. 4B). High expansions were obtained in NaOH 1N. The most expansive concrete is A (0.18% for A1 at 28 days vs 0.06% for B1; Fig. 4A). On the other hand, immersion in water resulted in very low expansions relatively to NaOH 1N (0.041% for A2 at 28 days vs 0.010% for B2). However, about 2 weeks after transfer to NaOH,

the sample A2, initially immersed in water, started to expand at the same initial rate as its companion A1, initially immersed in NaOH (Fig. 4B). This clearly indicates that NaOH penetrated quite easily in the cores, even for those with relatively low water/cement ratios (0.41 for A samples), thus increasing the amount of alkalies in the pore solution, and accelerating the reactions. Conversely, there is no doubt that the reverse effect (alkali dilution), also took place in the samples immersed in water, thus reducing, if not completely stopping, the reactions (after a certain period of time). In other words, the two conditions (NaOH 1N and water) are not realistic, and tests at 100% R.H. are certainly more appropriate, as previously discussed at section 3.4.

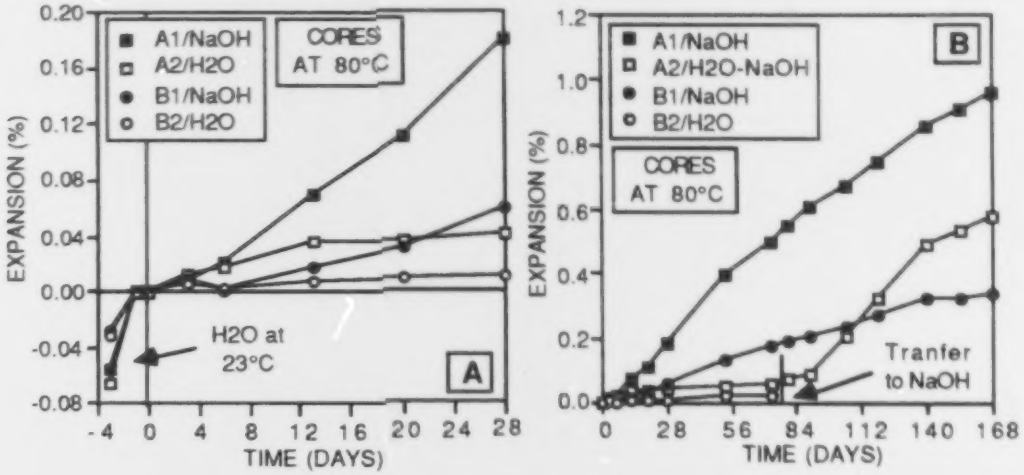

Fig.4 Expansion tests on two sets of companion cores (A1-A2, B1-B2) taken inside a 15 year old building from two concrete members. The samples were first immersed in water at 23°C until equilibrium (3 days), then transferred to water or NaOH 1N at 80°C (Fig. 4A). One core (A2) was transferred to NaOH after 73 days in water (Fig. 4B). (Note: On Fig. 4A, the thermal expansion that occurred during the first 24 hours at 80°C, has been substracted at time zero).

Interpretation. The limestone aggregates tested before (Fig. 1D), from Trois Rivières, are considered as the most reactive aggregates from the St.Lawrence Lowlands (Fournier & Bérubé 1989). Then how to explain why the laboratory concrete made with these aggregates expands significantly less in NaOH 1N at 80°C than concrete A, also containing limestone aggregates (Fig. 1C vs Fig. 4)? Indeed, the two concretes are very similar, except for the water/cement ratio (0.41 for A vs 0.55 for the laboratory concrete). However, at the NBRI Accelerated Mortar Bar Test (Oberholster & Davies 1986), a lower w/c ratio, at least down to 0.45, leads to lower expansions with limestone aggregates (Fournier & Bérubé 1990)! At least, the expansion tests in NaOH 1N suggest that the aggregates present in the concrete under test are moderately reactive (B) to very reactive (A), which is in good agreement with the petrographic examination. However, as mentioned above, this does not necessarily suggest that severe expansions are to be expected in the corresponding field concrete members. Indeed, the immersion solution is very concentrated in alkalies, and the humidity conditions are extreme. In the concrete components, these conditions could not be as severe. In the present case, water soluble alkali measurements are not available, but the total alkali contents supplied by the cements used are relatively low (2.9 kg/m^3 for A vs 1.8 for B). In fact, many reasons can be suggested to explain why concrete A is presently more deteriorated than concrete B: (1) a relatively higher alkali content, (2) a lower w/c ratio (the pore solution being much less

aboundant, then much more concentrated) (Bérubé & Fournier 1989), and (3) the aggregates are potentially more reactive, as suggested by the immersion tests in NaOH (higher amount of fine-grained siliceous limestone particles). On the other hand, the humidity conditions are thought to be sufficiently high to promote AAR at depths greater than a few centimetres inside the field concrete (see section 4). Based on all this information, it is concluded that the two concretes will continue to expand for a long time (at least 10 years), at about the same rates as presently, i.e. moderately fast for concrete A, and slowly for concrete B, which contains less alkalies and less reactive aggregate particles.

7. CONCLUSION

The laboratory expansion tests on core samples must be interpretated very carefully, in particular if they are performed in alkaline solutions (NaOH). In fact, such immersion tests are only useful to estimate the absolute residual degree of reactivity of the aggregates present in the concrete under test, since the experimental conditions are very severe with respect to alkalies and humidity. On the other hand, immersion in water should lead to alkali dilution in the pore concrete solution, and consequently to low expansions. Besides, the most popular method consists to perform the tests at 100% R.H. and 38°C.

In all cases, the results must be interpretated in accordance with the actual alkali content of the tested concrete, while the humidity conditions are thought to be sufficiently high inside most outdoor concrete members.This study demonstrated that common aggregates used in Quebec can release significant amounts of alkalies to the pore solution. More work is also required on the hot water extraction method proposed to measure the water soluble alkali content, in order to verify if the alkalies that have been progressively removed out of the pore solution, and entrapped in AAR products, are not also leached. If yes, this method should not be useful to determine the progress of the reactions.

According to Hobbs (1988): "It is clear that if expansion is observed (on cores), this does not necessarily mean that the actual concrete member from which the cores were taken will continue to expand. A more reliable procedure for establishing whether expansion is continuing is to monitor the actual movements of the structure and the affected concrete members. The potential for further expansion, if any, can then be estimated by extrapolation." Moreover, Palmer (1989) mention that the reproducibility and repeatability coefficients obtained from inter-laboratory trials on expansion tests on concrete cores, performed in accordance with BCA (1988), at 100% R.H. and 38°C, were too large for the test method to be positively recommended. Let us conclude with another sentence from the British Cement Association (BCA 1988) which confirms that the problem is not so simple, and need careful investigation and relevant experience: "Precise calculation of future reaction and expansion caused by ASR is not possible. It is however possible to produce a classification of the risk of future deterioration based on subjective assessment dependent on professional judgement backed by experience and expertise".

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REFERENCES

Al-Asali, M.M. & Malhotra (1989): V.M., Role of high-volume fly ash concrete in controlling expansion due to alkali-aggregate reaction. CANMET, Report MSL-89-66, 14 p., 1989.

Albert, P. & Raphael, S. (1987): Alkali-silica reactivity in the Beauharnois Power-Houses, Beauharnois. Concrete Alkali-Aggregate Reactions. Noyes Publications, 10-16. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Amasaki, S. & Takagi, N. (1989): The estimate for deterioration due to alkali-silica reaction by ultrasonic spectroscopy. Alkali-Aggregate Reaction - 8th International Conference. 839-844. (Proc. 8th ICAAR, Kyoto, Japan, 1989).

Barneyback, & Diamond, S. (1981): Expression and Analysis of Pore Fluids from Hardened Cement Paste and Mortar. Cement and Concrete Research, 11, 279-285.

BCA (British Cement Association) (1988): The Diagnosis of Alkali-Silica Reaction - Report of a Working Party. 36 p.

Bérubé, M.A., Frenette, J. & Fournier, B. (1988): Evaluation du potentiel résiduel d'expansion du béton de la centrale nucléaire de Gentilly II. Report GGL-88-25, Laval University, 23 p.

Bérubé, M.A. & Fournier, B. (1986): Les produits de la réaction alcalis-silice dans le béton: étude de cas de la région de Québec. Canadian Mineralogist, 24, 271-288.

Bérubé, M.A. & Fournier, B. (1989): Testing for Alkali-Aggregate Reactivity in Quebec. Proc. 1st Canadian Seminar on Cement and Concrete, Quebec City, 10 p.

Bérubé, M.A., Fournier, B., Pleau, R., Pigeon, M. & Frenette, J. (1989): Performance mécanique du béton atteint de réactivité alcaline. Paper presented at: Progress in Concrete 1989, ACI - Eastern Ontario and Quebec Section, Montreal, 43 p.

Blanchette, A. (1989): Evaluation du potentiel de réactivité alcaline des granulats à l'aide d'essais accélérés. Paper presented at: Progress in Concrete 1989, ACI - Eastern Ontario and Quebec Section, Montreal, 18 p.

Bouchard, M. (1987): Aménagement Beauharnois: mesure des déformations topographiques. Internal Report 302-93, Hydro Quebec.

Buil,M. & Zelwer, A. (1989): Extraction de la phase liquide des ciments durcis. Laboratoire Central des Ponts et Chaussées, Paris, 15 p.

Cavalcanti, A.J.C.T. & Silveira, J.F.A. (1989): Investigations on the Mixoto Powerhouse Concrete Affected by Alkali-Silica Reaction. Alkali-Aggregate Reaction - 8th International Conference. 797-802. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Charlwood, R.G. & Solymar, Z.V. (1989): AAR in Hydraulic Structures: a Global Overview. Proc. 20th Annual Acres Geotechnical Seminar, Niagara Falls, 40 p.

Diamond, S. (1989): ASR - Another Look at Mechanisms. Alkali-Aggregate Reaction - 8th International Conference. 83-94. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Fournier, B. & Bérubé, M.A. (1990): Application of the Accelerated Mortar Bar Test (NBRI Method) to the Carbonate Aggregates from the St.Lawrence Lowlands (Quebec, Canada). Submitted to Cement, Concrete and Aggregates.

Fournier, B. & Bérubé, M.A. (1989): Evaluation du potentiel de réactivité alcaline des granulats à béton produits dans les Basses-Terres du Saint-Laurent du Québec. Paper presented at: Progress in Concrete 1989, ACI - Eastern Ontario and Quebec Section, Montreal, 25 p.

Grattan-Bellew, P.E. (1981): Canadian Experience of Alkali-Expansivity in Concrete. Proc. 5th Int. Conf. on AAR, Cape-Town, South-Africa, Paper S252/6, 11p..

Hobbs, D.W. (1988): Alkali-Silica Reaction in Concrete. Thomas Telford Ltd., London, 183 p.

Ichihara, H., Shimamura, M. & Koshiishi, I. (1989): Alkali-Aggregate Reaction in Railway Prestressed Concrete Bridges. Alkali-Aggregate Reaction - 8th International Conference. 753-758. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Imai, H., Yamasaki, T., Maehara, H. & Miyagawa, T. (1987): The Deterioration by Alkali-Silica Reaction of Hanshin Expressway Concrete Structures - Investigation and Repair. Concrete Alkali-Aggregate Reaction. Noyes Publications, 131-135. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Kawakami, H. (1989): A Reinforced Concrete Building Deformed by Alkali-Aggregate Reaction. Alkali-Aggregate Reaction - 8th International Conference. 597-602. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Kawamura, M., Koike, M. & Nakano, K. (1989): Release of Alkalies from Reactive Andesitic Aggregates and Fly Ashes into Pore Solution in Mortars. Alkali-Aggregate Reaction - 8th International Conference. 271-278. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Kobayashi, K., Shiraki, R. & Kawai, K. (1989): Influence of Alkali Concentration Distribution Occurring in Concrete Members on Expansion and Cracking Due to Alkali-Silica Reaction. Alkali-Aggregate Reaction - 8th International Conference. 641-646. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Longuet, P., Burglen, L. & Zelwer, A. (1973): La phase liquide du ciment hydraté. Revue des Matériaux, 676, 35-41.

Mizumoto, Y., Kosa, K., Ono, K. & Nakano, K. (1987): Study of Cracking Damage of a Concrete Structure Due to Alkali-Silica Reaction. Concrete Alkali-Aggregate Reaction. Noyes Publications, 204-209. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Monfore, G.E. (1963): A Small Probe-Type Gage for Measuring Relative Humidity. PCA Research Department, 5, 2, 41-47.

Mukkerjee, P.K. & Bickley, J.A. (1987): Performance of Glass as Concrete Aggregates. Concrete Alkali-Aggregate Reaction. Noyes Publications, 36-42. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Nishibayashi, S. (1989): Alkali-Aggregate Reactivity in Japan - A Review. Alkali-Aggregate Reaction - 8th International Conference. 17-24. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Nixon, P.J. & Gillson, I.P. (1987): An Investigation into Alkali-Silica Reaction in Concrete Bases at an Electricity Substation at Drakelow Power Station, England. Concrete Alkali-Aggregate Reaction. Noyes Publications, 173-177. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Oberholster, R.E. (1983): Alkali Reactivity of Siliceous Rock Aggregates - Diagnosis of the Reaction, Testing of Cement and Aggregate and Prescription of Preventive Measures. Proc. 6th Int. Conf. on AAR, Copenhagen, Denmark, 419-433.

Oberholster, R.E. & Davies, G. (1986): An Accelerated Method for Testing the Potential Alkali Reactivity of Siliceous Aggregates. Cement and Concrete Research, 16, 181-189.

Okada, K., Utoh, S., Imai, H. & Ono, K. (1989): Concrete Structures Damaged by Alkali-Silica Reaction. Alkali-Aggregate Reaction - 8th International Conference. 791-796. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Olafsson, H. (1987): The Effect of Relative Humidity and Temperature on Alkali Expansion of Mortar Bars. Concrete Alkali-Aggregate Reaction. Noyes Publications, 461-465. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Palmer, D. (1989): Aspects of the Diagnosis of Alkali-Silica Reaction. Alkali-Aggregate Reaction - 8th International Conference. 741-746. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Rogers, C.A. (1987): Alkali-aggregate reactions in Ontario. Concrete Alkali-Aggregate Reaction. Noyes Publications, 5-9. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Rogers, C.A. & Hooton, R.D. (1989): Leaching of Alkalies in Alkali-Aggregate Reaction Testing. Alkali-Aggregate Reaction - 8th International Conference. 327-332. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Semmelink, C.J. (1981): Field Survey of the Extent of Cracking and Other Details of Concrete Structures Showing Deterioration Due to Alkali-Aggregate Reaction in the South Western Cape Province. Proc. 5th ICAAR, Cape-Town, South-Africa, Paper S252/19.

Seno, Y. & Kobayashi, K. (1989): Expansion Properties of Mortars and Variation in Components of Pore Solutions Due to Alkali-Aggregate Reaction. Alkali-Aggregate Reaction - 8th International Conference. 141-146. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Solymar, Z.V. (1989): Instrumentation and Monitoring of Concrete Expansion. Proc. 20th Annual Acres Geotechnical Seminar, Niagara Falls, 13 p.

Stark, D. (1984): Relative Humidity Measurements at Beauharnois Dam. Construction Technology Laboratories, Portland Cement Association, Report submitted to Hydro Québec, 11 p.

Stark, D. & DePuy, G. (1987): Alkali-Silica Reaction in Five Dams in Southwestern United States. Proc. Katherine & Bryant Mather Int. Conf. on Concrete Durability, Atlanta, USA, ACI SP-100, 1759-1786.

St-John, D.A. (1989): Alkali-Aggregate Reaction in New Zealand - A continuing Problem. Alkali-Aggregate Reaction - 8th International Conference. 51-56. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Tomita, M., Miyagawa, T. & Nakano, K. (1989): Basic Study for Diagnosis of Concrete Structures Affected by Alkali-Silica Reaction Using Drilled Concrete Cores. Alkali-Aggregate Reaction - 8th International Conference. 779-784. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Visvesvaraya, H.C., Rajkumar, C. & Mullick, A.K. (1987): Analysis of Distress Due to Alkali-Aggregate Reaction in Gallery Structures of a Concrete Dam. Concrete Alkali-Aggregate Reaction. Noyes Publications, 188-193. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Wakizaka, Y., Hirano, I., Kuwahara, K. & Makita, M. (1987): Characteristics of Aggregates in Concrete and Alkali-Silica Reactions in Japan. Concrete Alkali-Aggregate Reaction. Noyes Publications, 331-335. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Wang, H. & Gillott, J.E. (1989): The Effect of Superplastifiers on Alkali-Silica Reactivity. Alkali-Aggregate Reaction - 8th International Conference. 187-192. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Wood, J.G.M. (1985): Alkali-Silica Reaction - Concrete Society Conference. Engineering Assessment of Structures with ASR, 8 p.

Wood, J.G.M., Young, J.S. & Ward, D.E. (1987): The Structural Effects of Alkali-Aggregate Reaction on Reinforced Concrete. Concrete Alkali-Aggregate Reaction. Noyes Publications, 157-162. (Proc. 7th ICAAR, Ottawa, Canada, 1986).

Wood, J.G.M., Norris, P. & Leek, D. (1989): Physical Behavior of AAR Damaged Concrete in Structures and in Test Conditions. Alkali-Aggregate Reaction - 8th International Conference. 765-770. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Wroblewicz, Z., Solymar, Z. & Thompson, G.A. (1988): Deformation Monitoring Instrumentation at Mactaquac Generating Station, New Brunswick, Canada. Proc. 5th Int. (FIG) Symp. on Deformation Measurement, Fredericton, Canada, 593-601.

Yonezawa, T., Ashworth, V. & Procter, R.P.M. (1989): The Mechanism of Fixing Cl^- by Cement Hydrates Resulting in the Transformation of NaCl to NaOH. Alkali-Aggregate Reaction - 8th International Conference. 765-770. (Proc. 8th ICAAR, Kyoto, Japan 1989).

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

CASE STUDIES OF ONTARIO HYDRO'S EXPERIENCE WITH STANDARD TESTS FOR ALKALI-AGGREGATE REACTIVITY

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ABSTRACT

The results of laboratory test methods to pre-test aggregates and aggregate-cement combinations for potentially deleterious alkali-aggregate reactivity are not always consistent. It is always preferable to perform as many of the test methods as possible to help in analysing results. In some cases, a deleteriously reactive aggregate may pass one or more of the tests, and in other, less frequent cases, good aggregates can fail a test. The best indicator of an aggregate's durability is previous field experience or a survey of existing structures made with similar aggregate-cement combinations in a moist environment. In this paper, several case studies of anomalous laboratory tests with Ontario aggregates are described.

1. BACKGROUND

Evaluation of Ontario Hydro's project aggregates for potential alkali-aggregate reactivity was initiated by R.B. Young within months of Stanton's original paper [1] which explained the mechanisms responsible for the deleterious expansion. Subsequently, extensive series of what later became the ASTM C 227 mortar expansion test were conducted for most of the hydraulic projects including the St. Lawrence power development. In addition to petrographic analysis under the supervision of Ludmilla Dolar-Mantuani, other test methods were conducted as they were proposed and later adopted by ASTM and CSA standards such as the ASTM C 289 Quick chemical Method, ASTM C 342 Conrow test and the CSA CAN-3-A23.2-14A Concrete prism test.

2. TEST METHODS

In addition to petrographic analysis, in North America there are several standard test methods for detecting potentially deleterious alkali-reactive aggregates or aggregate cement combinations. a very brief outline of the more common ones are given here.

2.1 ASTM C 227

Aggregates are crushed to sand sizes and mortar bars are cast. After stripping the molds at one day, the bars are placed into sealed containers over water to maintain a 100% humidity environment and stored at 38°C to accelerate any potential reaction. Expansions need to be measured for 6 months (by the ASTM C 33 suggested criteria of 0.10% at 6 month) but it is advisable to monitor expansions for at least one year and experience has shown that 2 years is required for some of the slowly reactive aggregates to expand. Although this test is the most widely used worldwide, care must be taken in container design and handling since the bars are sensitive to leaching of alkalies, which stops the reaction [2].

2.2 ASTM C 289

Known as the quick chemical test, a sample of crushed aggregate is placed in a container with 1N NaOH solution and reacted for 24 hours at 80°C. the quantity of dissolved silica (from the aggregate) and the reduction in alkalinity (due to reactions using up the NaOH) are measured and plotted. Based on experience, boundaries have been developed to classify the aggregates as either innocuous, potentially deleterious or deleterious.

2.3 ASTM C 342

In the Conrow test, mortar bars are prepared as in ASTM C 227. Then the bars undergo an unusual cycle with storage in water at 23°C until 28 days, heating to 55°C in water for 7 days, cooling in water to 23°C for 1 day, heating in air at 55°C for 7 days then replacement in water at 23°C for up to 1 year. An expansion (from 48 hours of age to 1 year) of more than 0.20% is considered unacceptable.

2.4 CSA CAN3-A23.2-14A

In the Swenson test, concrete prisms are cast and placed in either a moist room (standard, 23°C test) or in a sealed container over water at 38°C (accelerated, 38°C test). The standard mix has a cement concrete of 310 kg/m^3 and an alkali content of 1.0% by mass of cement (in the 1986 revision, this was raised to 1.25%).

Expansions are measured for up to one year. The recently adopted CSA suggested expansion limits are 0.01% at 3 months and 0.025% at one year for 23°C storage, and 0.04% at 1 year for 38°C storage [3].

3. CASE STUDIES

3.1 Alkali-Carbonate Reactivity

In the 1950's, Ontario Hydro developed its own quarry in Cornwall for construction of Saunders GS. on the St. Lawrence River. At that time, the alkali-carbonate reaction was unknown and Hydro was fortunate that only the non-reactive upper lifts of limestone were excavated. Exhaustive series of ASTM C 227 mortar bars were carried out with various combinations of aggregates (to detect a possible pessimum effect) but, as was later found by Swenson [4], mortar bar testing would not have detected the problem. Many years later, after Swenson had proposed a concrete prism test, concrete prisms were made and stored at both 23°C and 38°C, which confirmed that the aggregate was not alkali-carbonate reactive. Fortunately the only Ontario Hydro structure known

to be afflicted by the alkali-carbonate reaction was a small transformer sub-station constructed in the 1950's near Kingston, Ontario.

3.2 Lower Notch G.S. and Lady Evelyn Lake Dam

In 1965, prior to the construction of Lower Notch Generating Station (G.S.), at the mouth of the Montreal River, (south of Cobalt on the Quebec border), the old (1925) Lady Evelyn Lake control dam further up the river was found to be in severe distress. Some of the piers had been cracked and severely tilted by expansion of the deck sections. Mortar bars (Figure 1) and concrete prisms made from rocks in the local outcrop were found to expand slowly but deleteriously (i.e. greater than 0.10%, and accompanied by cracking). Subsequent petrographic analysis showed these aggregates to be mostly argillite-greywackes and concrete cores from the dam exhibited an unusual alkali-silica reaction. Not much gel was produced and the aggregates cracked internally then spread into the paste. Further study by Dr. Dolar Mantuani [5] made the old Lady Evelyn Lake dam internationally famous and indicated that many other old structures in the area were also deteriorating and that the reactive rock in the Huronion Supergroup extended south-west to Sudbury and beyond (Figure 1).

The concern was that C 227 mortar bars made with this aggregate did not exceed the ASTM and CSA expansion limit of 0.10% at 6 months or in the case of Lower Notch, the more stringent U.S. Bureau of Reclamation limit of 0.10% at one year. Expansions exceeding the generally accepted critical 0.10% value were only obtained after 12 to 24 months of storage at 38°C. The CSA has now recognized this as the slowly expansive alkali-silicate reaction [3]. The Lady Evelyn Lake dam was damaged to the point that it was removed and replaced with a new dam (using low alkali portland cement) in 1973. Visual inspection and cores taken in 1985 indicate that the concrete in the new dam is in good condition. Therefore, at Lower Notch G.S, where the fore-bay excavation rock (70% greywacke with a fine grained argillite matrix) was to be used for concrete aggregate, alkali-aggregate testing was initiated well in advance of construction. ASTM C 227 mortar bar tests (Figure 3) and 38°C concrete prism tests (Figure 4) showed that this rock was also slowly reactive (meeting the ASTM C 227 limit) and that 20% replacement of normal, high-alkali portland cement (NPC) by a class F fly ash (FA) gave equivalent reductions in expansions to either low-alkali (LAPC) or low-alkali, sulphate-resistant (SRPC) portland cements. The ASTM C 289 quick chemical test classified this rock as innocuous and therefore, was inaccurate for this type of reactive rock. However, the Lady Evelyn Lake, aggregate contained more coarsely grained argillites and was classified as potentially reactive by C 289.

Because of the pre-testing, the concrete specification for Lower Notch GS included precautions and allowed a choice of either low-alkali cement or a combination of normal cement and fly ash. Partially because of a shortage of low-alkali and low-heat cements, the fly ash option was chosen (20% replacement in structural concrete and 30% in mass concrete) and the concrete in this dam which was completed in 1969 is performing well based on visual inspection and cores taken in 1985. As well, it is probably the only hydraulic structure in North America where fly ash was intentionally used to control an alkali-reactive aggregate [6].

3.3 Aubrey Falls GS

This generating station was built between 1967 and 1969 on the Mississagi River (also shown in Figure 2). The petrographic analysis of the concrete sand from Miller's pit indicated that it was composed of approximately 75% quartz and feldspar grains and aggregates (with 10% aplite and gneiss; 13% diabase, greenschist and volcanics). While this sand was petrographically acceptable

with only 1.5 to 2.5% of unsound particles, concern was raised because of the then current literature about altered/weathered feldspars causing expansion in concrete in Kansas, Iowa, Oklahoma and Nebraska [7]. There the C 227 test did not detect a problem but a new test, involving initial wet and dry cycling of mortar bars was developed by Conrow [7] and later adopted as ASTM C 342. However, the sand for Aubrey Falls was almost petrographically identical to that used to build the nearby Rocky Island control dam in the late 1940's and which was performing well (visual inspection of this non-air entrained dam in 1985 showed it to be in excellent condition, with the exception of local freeze/thaw damage due to leaking joints on the downstream face of the gravity section). As well, neither the ASTM C 227 mortar bar test nor the C 289 quick chemical test indicated an alkali-aggregate problem. Unfortunately, the new C 342 Conrow test was carried out and deleterious expansions (in excess of 0.6%) were obtained as shown in Figure 5. Even cement having only 0.61% Na_2O eq. resulted in expansions exceeding the ASTM 0.2% limit at one year and only 20% fly ash replacement totally prevented expansion in this test (although a 0.35% Na_2O eq. cement did pass the test). Interestingly, a modified version of the Conrow test was carried out using concrete prisms instead of mortar bars and none of the expansions exceeded 0.007%. Eventually, Aubrey Falls was built with this sand using high alkali normal portland cement and fly ash. (The stone was mainly granite with Nippissing diabase dykes and feldspars). However, due to this case and similar anomalous performances of the Conrow test in other areas of North America, the ASTM C 342 test now states that it is only applicable for specific aggregates in the states of Kansas, Nebraska, Oklahoma and Iowa.

3.4 Rejected Thede Gravel

Gravel from the Thede pit in south western Ontario was rejected for water line concrete at Bruce GS because it contained 5% leached chert which would be unacceptable due to poor freeze/thaw performance (similar, leached cherts are responsible for pop-outs in a wide range of concrete in S.W. Ontario.). The rest of the gravel was composed of 75% carbonate and 20% siliceous rocks.

Ontario Hydro's Research Division initiated a study of this aggregate as part of its long-term studies of aggregate performance. The C 289 quick chemical test indicated that this aggregate was innocuous but close to the borderline. The C 227 mortar bar expansions were innocuous (0.028% at 1 year and 0.039% at 8 years) as were the 23°C concrete prism expansions (0.007% at 12 years). In addition, two 300 × 300 × 450 mm blocks which were semi-immersed in water at the Research Division's Outdoor Exposure Site had actually contracted by 0.006% and 0.012% after 12 years and visually are in good condition.

However, additional experiments were conducted using added NaOH to accelerate potential alkali-silica expansions raising the alkali content of C 227 mortar bars by 2.0% to approximately 3% Na_2O eq. These bars expanded deleteriously (0.167% at 1 year) except when 20% fly ash replacement was used (0.032% at 1 year) (Figure 6). As a control test, Ontario Hydro's Laboratory standard Paris sand and gravel (both of which also contain a fraction of one percent chert, and might be considered petrographically marginal but with a long history of good field performance in concrete) were also made into mortar bars with 2.0% added alkali. Again the 0.10% expansion limit at one year was exceeded unless fly ash replacements were included (Figure 7).

Therefore, it appears that the use of high alkali conditions to accelerate alkali-silica tests may cause petrographically marginal, but satisfactory aggregates to be rejected and need to be viewed with caution.

4. SUMMARY AND CONCLUSIONS

1. If properly carried out with precautions to prevent alkali-leaching, the ASTM C 227 mortar test is usually reliable in terms of causing reactive aggregates to expand. However, the test is slow and with the argillite/greywacke rock in Northern Ontario, the ASTM and CSA failure limits are inadequate. Since the Lady Evelyn Lake dam study, Ontario Hydro has tightened its own expansion limits for specific projects.

2. The ASTM C 289 quick chemical test has been found to be lacking in terms of reliability and it has been discontinued by Ontario Hydro.

3. The ASTM C 342 Conrow mortar bar test was found to falsely consider a feldspar sand reactive. This ASTM method has since been modified to warn against its use outside of Oklahoma, Kansas , Nebraska and Iowa and is no longer considered for use in Ontario.

4. The CSA A23.2-14A Concrete prism test at 38°C, which was developed to detect the alkali-carbonate reaction, appears also to be valid for evaluating the slowly expansive alkali silicate reaction.

5. The use of added alkali to accelerate the rate of expansion of mortar bars should be viewed with caution especially with the chert-containing, petrographically marginal aggregates in South Western Ontario.

6. Examination of existing structures made with similar aggregates is still one of the best ways to evaluate potential alkali-aggregate reactivity.

REFERENCES

[1] Stanton, T.E., Expansion of Concrete Through Reaction Between Cement and Aggregate, Proceedings, ASCE, 66, 1781, 1940.

[2] Rogers, C.A. and Hooton, R.D., Leaching of Alkalies in Alkali-Aggregate Reaction Testing, Proceedings, Eighth International Conference on Alkali-Aggregate Reaction, Kyoto, 327, 1989.

[3] Canadian Standards Association, Appendix B - Alkali-aggregate Reaction, Supplement No. 2 to CAN3-A23.1-M77, 37 pp., 1986.

[4] Swenson, E.G., A Reactive Aggregate Undetected by ASTM Tests. ASTM Bulletin, 226, 48, 1957.

[5] Dolar Mantuani, L., Alkali-Silica-Reactive Rocks in the Canadian Shield, Highway Research, Record No. 268, 99, 1969.

[6] Mather, B., Personal Communication, Chief, Structures Laboratory, US Army, Waterways Experiment Station, 1985.

[7] Conrow, A.D., Studies of Abnormal Expansion of Portland Cement Concrete, Proceedings, ASTM, 52, 1205, 1952.

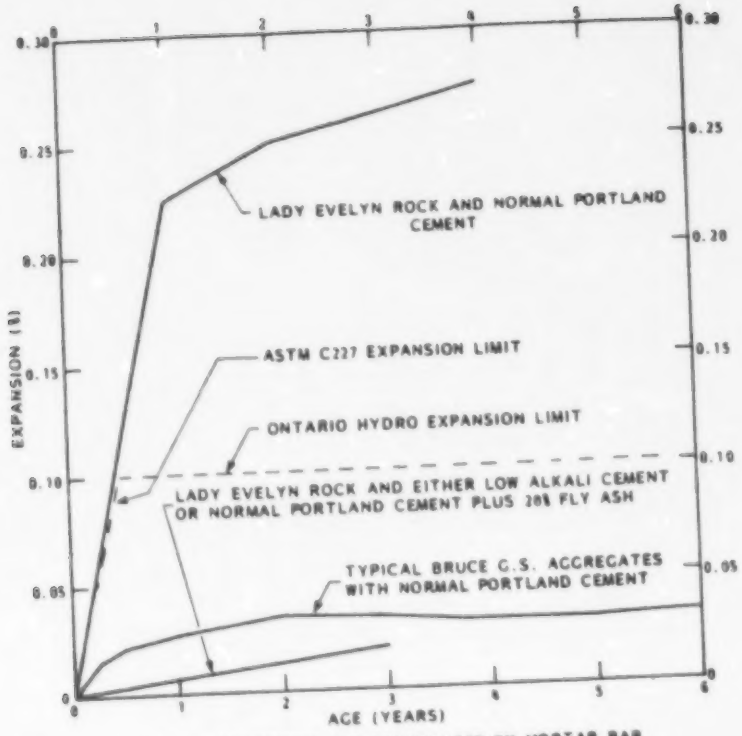

EXPANSION (%)
AGE (YEARS)
LADY EVELYN ROCK AND NORMAL PORTLAND CEMENT
ASTM C227 EXPANSION LIMIT
ONTARIO HYDRO EXPANSION LIMIT
LADY EVELYN ROCK AND EITHER LOW ALKALI CEMENT OR NORMAL PORTLAND CEMENT PLUS 20% FLY ASH
TYPICAL BRUCE G.S. AGGREGATES WITH NORMAL PORTLAND CEMENT

Figure 1: ALKALI REACTIVITY OF AGGREGATES BY MORTAR BAR EXPANSIONS AT 38°C AND 100%RH (ASTM C227)

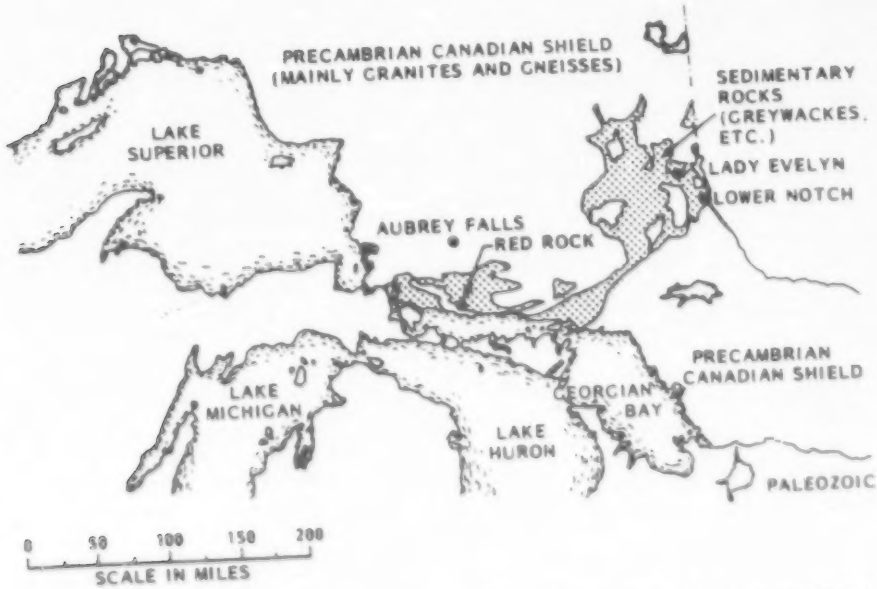
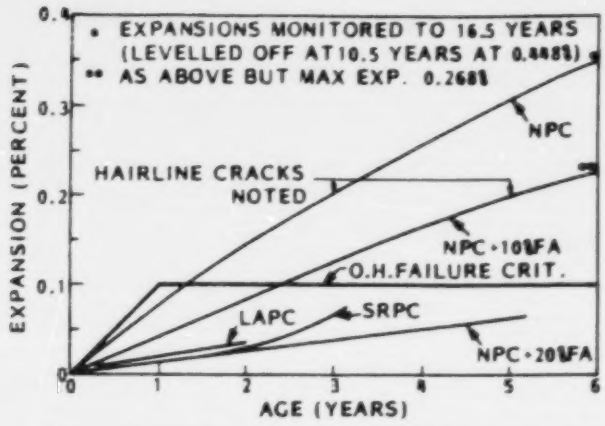


Figure 2: COMPARISON OF COMPOSITIONS OF SAND-GRAVEL DEPOSITS IN THE CANADIAN SHIELD



MAT'LS	K_2O	Na_2O	Na_2O EQUIV.
NPC	0.85	0.37	0.93
LAPC	0.37	0.11	0.35
SRPC	0.58	0.01	0.38
FLY ASH	DETROIT EDISON, LOI = 4.16%		

Figure 3: ASTM C227 MORTAR BAR EXPANSIONS-GREYWACKE WITH ARGILLITES CRUSHED ROCK-LOWER NOTCH GS - EFFECTS OF CEMENT AND FLY ASH

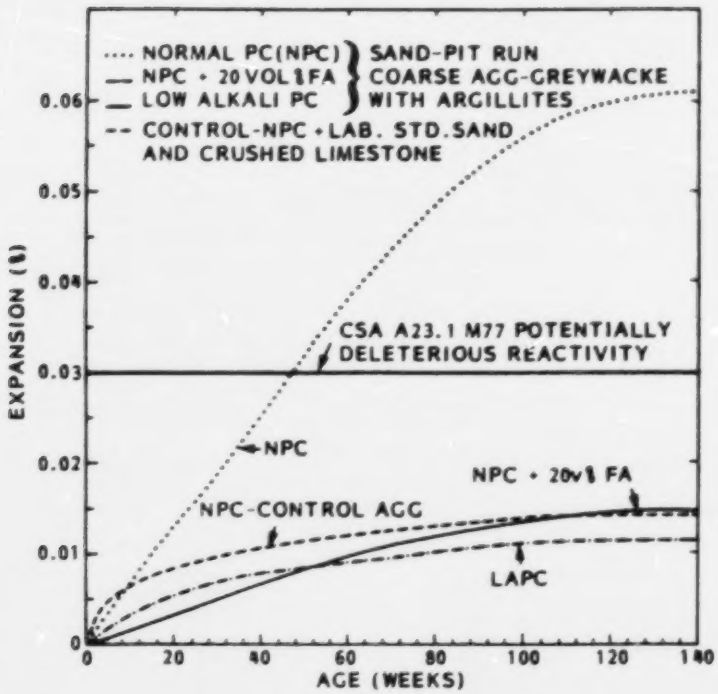
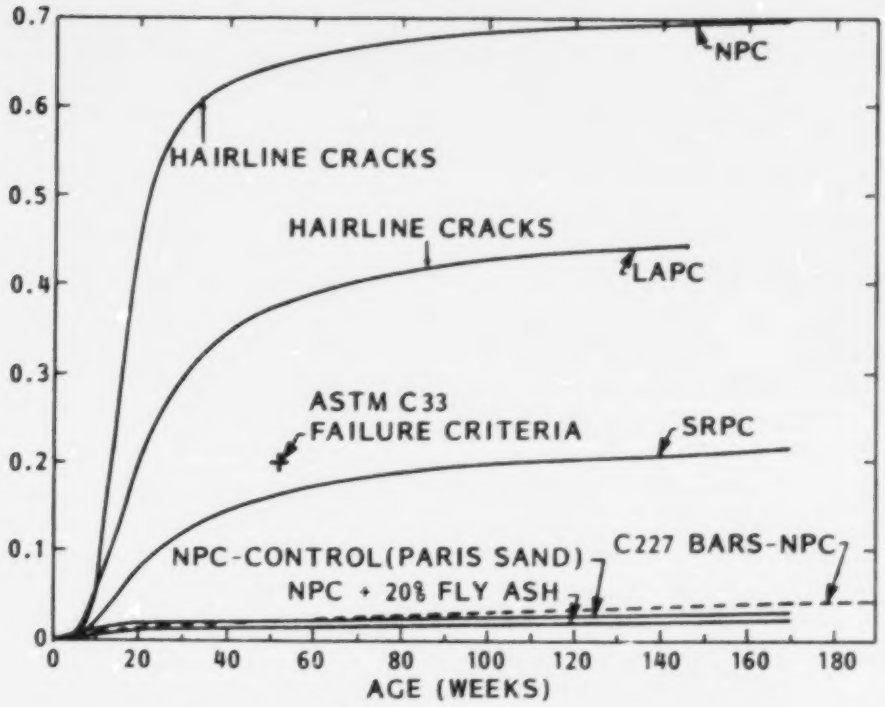


Figure 4: 38°C CONCRETE PRISM EXPANSIONS - LOWER NOTCH GS 1968-71 (NOW CSA A23.2-14A METHOD)



LEGEND		Na_2O EQUIV.
NPC	- NORMAL PORTLAND CEMENT	1.16%
LAPC	- LOW ALKALI P.C.	~0.61%
SRPC	- SULPHATE RESISTANT P.C.	0.35%
FLY ASH	- DETROIT EDISON LOI = 4.1%	

Figure 5: ASTM C342 CONROW MORTAR BAR EXPANSIONS USING FELDSPAR RICH "MILLER" SAND AUBREY FALLS G.S.

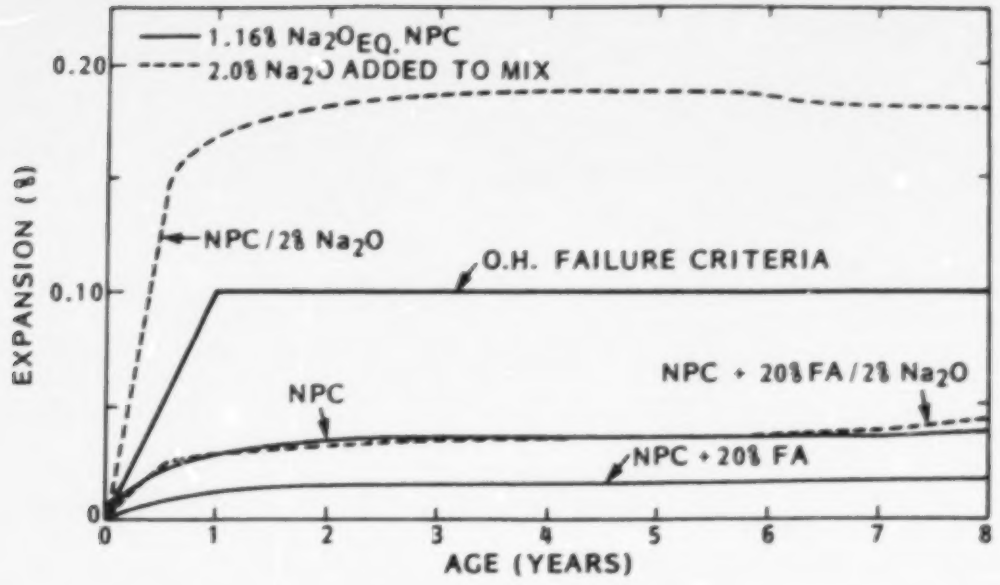


Figure 6: ASTM C227 MORTAR BAR EXPANSIONS - WITH REJECTED CRUSHED GRAVEL (GRAVEL REJECTED DUE TO 5% CHERT)-EFFECT OF ADDED Na_2O AND FLY ASH

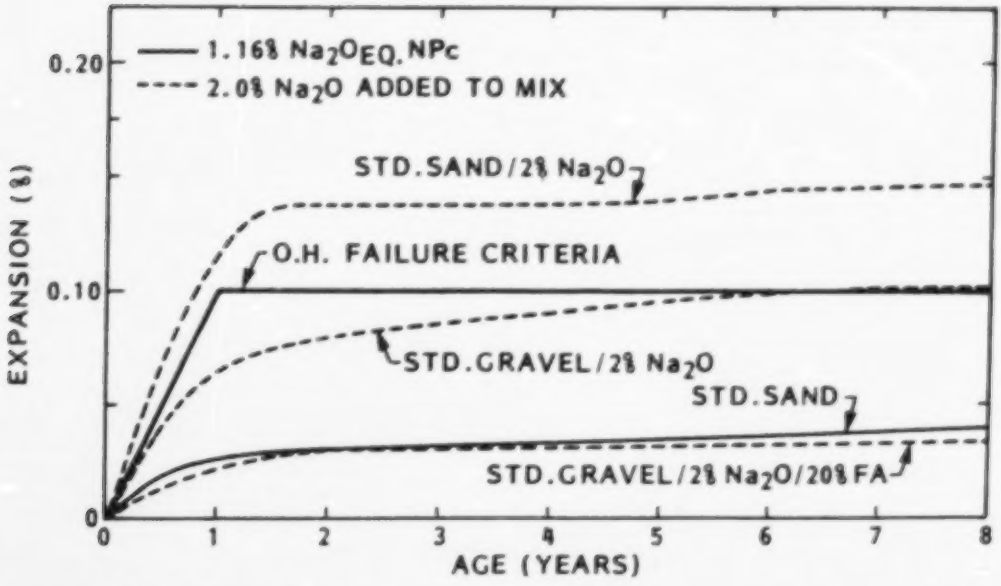


Figure 7: ASTM C227 MORTAR BAR EXPANSIONS WITH LAB. STANDARD SAND AND GRAVEL - EFFECTS OF ADDED Na_2O AND FLY ASH

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

STORAGE CONDITIONS AND RELIABILITY OF THE ASTM C227 MORTAR BAR AND CSA A 23. 2-14 A CONCRETE PRISM TESTS

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1. INTRODUCTION

When a Research Group enters a new area dealing with practical problems, such as those of durability, He legitimately wants the results to be based on reliable, reproducible and, if possible, rapid tests.

This is particularly true in the AAR field, for which the situation has been up-dated during the 8th ICAAR (Kyoto), in july 1989 :

- On one hand, some current studies show a trend towards the development of accelerated tests, giving an answer within a few days. This is precisely the subject of Hooton and Rogers paper [8] : Evaluation of rapid test methods for detecting alkali-reactive aggregates.

For most of them however, the quantity of available results is not yet sufficient, and more work is needed : this is mentioned many times in P.G Bellew's Keynote lecture [1].

- On the other hand, and here I am quoting the introduction of this paper "test methods such as the mortar bar method (ASTM C227) and the concrete prism test method (CSA A 23.2 - 14 A) which have traditionally been used to evaluate the potential reactivity of aggregates are no longer considered satisfactory... they take too long to complete... there is a need to refine existing standard tests..., reliable criteria for the evaluation also need to be improved."

This author summarizes quite well the situation which we found when we started work in the AAR field, three years ago.

Our choice has been to begin with conventional standards, rather than with accelerated tests, although we are of course interested in these and work with them.

In other words if the three conditions : reliable, reproducible and rapid cannot be met at once, then we prefer to have reliable and reproducible results.

Therefore, we have conducted a large programme in order to determine to what extent the standard mortar bar method ASTM C227 and the Canadian concrete prism test method CSA A 23.2-14 A are reliable and reproducible. The presentation and discussion of the results obtained in improved preparations and storage conditions, are the main purposes of the present paper. Part of them have already been given in our paper presented at the Kyoto conference [2].

2. MATERIALS

Following the same logic, we have decided to work with real, natural aggregates, known to develop AAR in existing structures. Thus, three Canadian aggregates, representative of the three types of reactions and considered as standards, have been chosen [3]. They are available from stock piles, through the Ministry of Transportation of Ontario.

As the data concerning these aggregates are periodically up-dated in a booklet called : General information on standard alkali-reactive aggregates from Ontario (last issue : 1988), their presentation will be brief and only the necessary information will be given, such as the chemical analysis, for the Spratt and the Sudbury aggregates : Table 1

TABLE 1 : Chemical analysis of Spratt and Sudbury aggregates

Aggregate	SiO2	TiO2	Al2O3	Fe2O3	CaO	MgO	SO3 T	L.O.I	K2O	Na2O	Cl
Spratt (A.R)	8.9	-	0.6	0.30	48.85	1.45	0.30	39.65	0.09	0.03	-
Spratt (Ins.R)	86.5	-	4.2	1.45	1.50	0	3.15	1.85	0.56	0.10	-
Sudbury	69.25	0.65	13.00	4.40	1.75	3.10	0.15	1.80	1.95	3,18	0.005

This paper does not deal with the dolomitic Pittsburg aggregate.

- see our parallel paper for details concerning the alkali silica reactive Spratt aggregate,

- the Sudbury aggregate, which is a partly crushed gravel from a pit near Sudbury airport, is representative of a slow alkali-silicate reaction. Used in over 26 highway structures, nearly all of them showed damage due to AAR. The gravel contains about 75 % reactive particles : argillite, greywacke, sandstone, arkosic sandstone, quartz wacke and quartzite, among which argillite is the most reactive one, but, probably more important, the composition and microstructure of the reactive materials (illite and micro crystalline quartz are similar in all the constituents of the aggregate [4].

- The cement used for all the studies is a normal Portland cement of ≥ 0.75 % Na_2O equivalent alkalies.

3. THE ASTM C227 MORTAR BAR METHOD

3.1 The Major Problems and Initial Solutions

Several recent papers have disturbed the opinion about this method ; the most striking one is probably the Oberholster's paper reporting the data of an Interlaboratory testing program [5] : out of 5 reactive aggregates in field conditions, 4 were shown innocuous with

the mortar bar test !

Among the problems discussed in the litterature (Cf. ref. 1.), the following factors appeared to us of critical importance :

- the alkali level [6]. Following a series of disappointments we have decided to use a mortar containing 1.25 % Na2O eqv., as now prescribed in the Canadian concrete prism method. This is obtained by adding NaOH to the mixing water.

- the influence of the storage conditions [7] and more specifically : the achievement and maintenance of the 100 % RH and 38°C.

3.2 Selection of the appropriate Storage Conditions using Spratt's aggregate

A specific chapter is devoted to detail the equipment, and its operating mode (chapter 4), so, refer to it and to figures 1 and 2 for a better comprehension of this part of the program.

Three storage conditions have been tested :

A - The metallic container is placed inside the reactor, at 38°C and 100 % R.H.

B - Same as A, except that a wick lines the internal walls of the reactor.

C - The specimens are placed in a polyethylene bag containing a small quantity of water. In order to prevent the bars from being in contact with water, they are supported on a plastic stand.

To test the reproductibility of the method, in the three modes, the experimental program has been conducted in the following manner : three series of four bars have been prepared at one month intervals by three operators ; the corresponding results are in lines 1, 2 and 3 in table 2. Each value is the mean of the 4 individual bars.

Table 2 : Testing the appropriate storage conditions with Spratt's aggregate

Storage conditions		Expansion (%)						
		1 m	2 m	3 m	6 m	8 m	10 m	12 m
A	1	0.023	0.078	0.137	0.284	0.299	0.309	0.320
	2	0.026	0.084	0.165	0.306	0.328	0.344	0.350
	3	0.020	0.109	0.182	0.263	0.297	0.303	0.311
B	1	0.021	0.071	0.118	0.229	0.247	0.250	0.255
	2	0.026	0.079	0.155	0.261	0.272	0.287	0.291
	3	0.021	0.112	0.184	0.258	0.281	0.286	0.295
C	1	0.024	0.091	0.149	0.286	0.305	0.325	0.352
	2	0.027	0.094	0.165	0.315	0.324	0.363	0.332
	3	0.019	0.102	0.173	0.268	0.297	0.308	0.320

Whatever the mode tested (A, B or C), the results are reproducible. However, as mode A gives slightly better results and is also experimentally the simplest one, it has been selected as

the most appropriate, and further testing has been done according to mode A.

We have two comments here :
1 - Results according to mode A are close to those obtained with the old hydro 4 bar (no wicks) reported in the Ontario Hydro MTC study of ASTM C227 containers, with Spratt's aggregate (cement Na_2O eqv. : 1.17 %) [7]. The results of this study are worth being considered. Out of the nine types of container, 3 only rated the Spratt aggregate as reactive, and only the one mentioned above (hydro 4 bar) correctly reflected it's expansion potentiality.

2 - Testing 12 aggregates with 2 storage modes (3 of the 6 bars are stored upright at 100 % RH, 38°C in wicked, rectangular stainless steel container ; the remaining 3 are placed in heat-sealed, polyethylene bags with approximately 10 ml of water), Rogers and Hooton found that only the bars that were sealed in bags expanded at all [7].

We are convinced, from results on mortar bars (Table 2) as well as those obtained with the concrete prisms (Table 4), that the problem is not whether to use plastic bags or not, but is much more related with a good homogeneity of temperature and relative humidity (within the reactor and as a function of the time).

3.3 Results with Sudbury aggregate

Three series of 4 bars have been stored, following mode A. The results, wich appear in Table 3, show that the reproducibility is good, but confirm previous findings that this aggregate is not classified as reactive, even when considering the revised limits of expansion : 0.05 % at 6 months and 0.10 % at 12 months, as suggested by many agencies including Ontario Hydro, U.S. Army and Bureau of Reclamations. With this late-expanding alkali silicate reaction aggregate the 0.10 % expansion can be expected between 18 and 24 months. The test must therefore be extended or a second possibility is to revise the limit of expansion at 6 and 12 months.

Table 3 : Mortar bars expansion data for Sudbury aggregate

Mode A	Expansion (%)							
	1 m	2 m	3 m	4 m	6 m	8 m	10 m	12 m
1	0.018	0.020	0.024	0.030	0.040	0.049	0.053	0.057
2	0.007	0.013	0.017	0.023	0.034	0.042	0.048	0.051
3	0.012	0.013	0.020	0.022	0.032	0.038	0.044	0.047

4. SPECIFIC APPARATUS

Taking into account the results in publications and our own results, we have paid a great deal of attention to the quality of the test conditions : 100 % RH and 38°C. This means : invariability as a function of the time as well as -this is essential- homogeneous repartition along a given bar and from one bar to another. In many containers, this is not the case. To meet these conditions the equipment shown in Figures 1 and 2 has been designed.

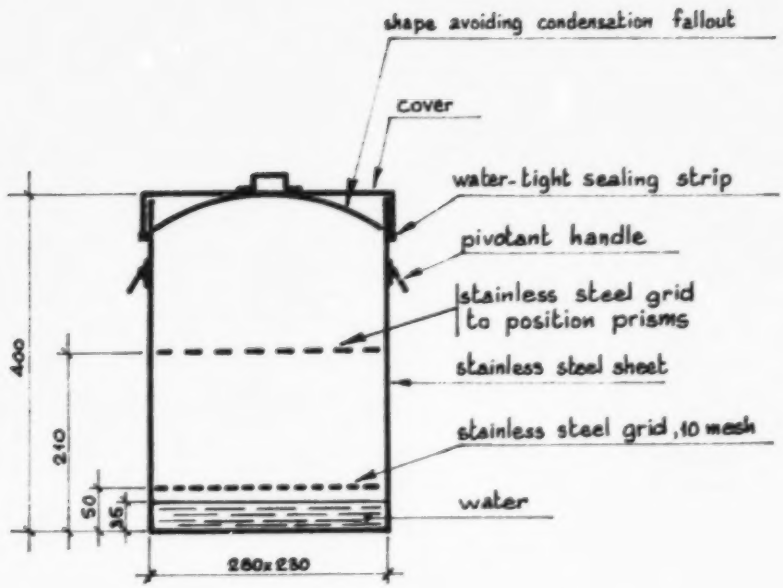

Figure 1 : The container with a specially designed cover is able to receive 30 mortar bars or 6 concrete prisms. They stand upright on the lower stainless steel grid.

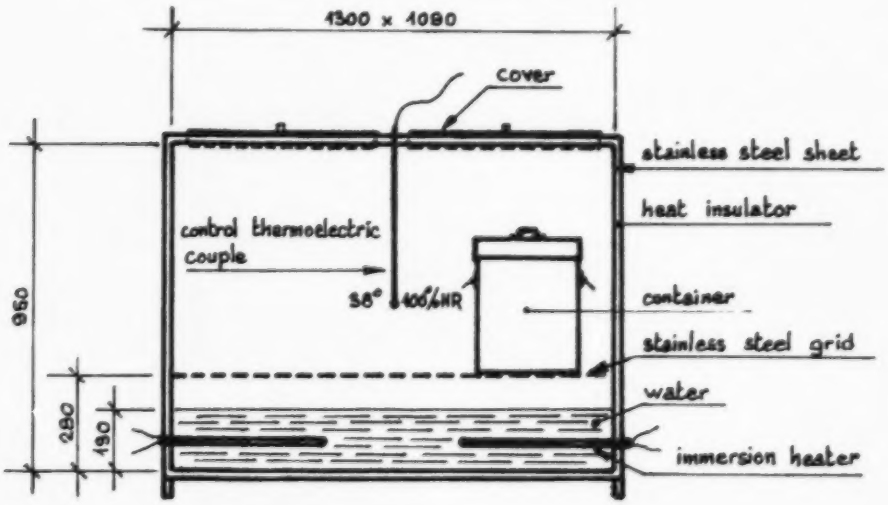

Figure 2 : The metallic reactor may contain 9 containers. The double regulation system ensures the homogeneous repartition and maintenance of the 100 % RH and 38°C.

This device has been set up for the ASTM C227 mortar bar test, as well as for the Canadian concrete prism test ; the only difference is the number of specimens stored in each container.

The conception of the equipment is based on the principle of a double regulation system of temperature and R.H. : the container with water in the bottom is placed in a large box, thermoregulated at 38°C and itself containing some water. This equipment is detailed in a separate paper [9].

We may add that the container, made of stainless steel, has a suitably designed cover in order to avoid alkali leaching.

5. THE CONCRETE PRISM TEST METHOD (CSA A 23. 2 - 14 A)

5.1 Determination of the Best Storage Conditions, using Spratt's Aggregate

The problems of reliability and reproducibility were also the first ones to be investigated : the experimental programme has been conducted in the same manner as the programme established for the mortar bar test ; the letters A, B, C and numbers 1, 2, 3 keep the same significance.

1.25 % Na_2O eqv. has also been used here and NaOH has been added to the mixing water. The revised standard method prescribes this level of 1.25 % Na_2O eqv. Previous works have shown that a too low content of alkalies fail to detect reactive aggregates, for examples : rhyolitic tufs and limestones from the Ottawa or Trois Rivières regions [10].

A non reactive sand (pure limestone) has been taken as fine aggregate. For this part of the program the cement content was 310 kg/m3 ; a special study has been devoted to the influence of the cement content (section 5.3).

The results appear in table 4 :

Table 4. Determination of the most appropriate storage conditions of the concrete prisms, using Spratt's aggregate

STORAGE CONDITIONS		Expansion (%)					
		2 m	3 m	6 m	8 m	10 m	12 m
A	1	0.073	0.099	0.148	0.159	0.170	0.177
	2	0.032	0.066	0.108	0.123	0.131	0.138
	3	0.040	0.070	0.117	0.129	0.134	0.136
B	1	0.066	0.098	0.139	0.150	0.156	0.162
	2	0.031	0.065	0.113	0.123	0.131	0.138
	3	0.033	0.066	0.107	0.113	0.117	0.117
C	1	0.027	0.050	0.088	0.103	0.113	0.126
	2	0.022	0.038	0.060	0.077	0.078	0.099
	3	0.016	0.043	0.081	0.091	0.099	0.109

The method A appears superior, and results are even clearer than those of the mortar bar

method. Therefore method A was definitively adopted for both tests.

As early as 2 months, the expansion reaches the prescribed limit (0. 04 %) for categories A, B and C, and there is no ambiguity in classifying this aggregate as reactive. It was of course interesting to test our selected conditions with the more problematic, slow expanding, Sudbury aggregate.

5. 2 Testing the Validity of the Selected Method (A) with Sudbury Aggregate

It is important to note, that with a 310 kg/m3 cement content, the Sudbury aggregate has reached the limit of reactivity at 10 months.

Table 5 : Behavior of the Sudbury aggregate when tested in concrete prisms (310 kg/m3 cement content)

Expansion (%)							
1 m	2 m	3 m	4 m	6 m	8 m	10 m	12 m
0	0.001	0.003	0.007	0.014	0.027	0.046	0.063

Our results agree with previous ones on the following point that the concrete prism method is more appropriate than the ASTM C227 mortar bar test to show the reactivity potential of such slowly expanding silicate aggregates [4]. They also show that there is no absolute need to increase the cement content to reach the expansion limit within the allowed time of reaction (12 months).

5.3 Influence of the Cement Content

Even if we can classify as reactive the slowly expanding aggregates with a 310 kg/m3 cement content, it is nevertheless worth observing the influence of a higher cement content (410 kg/m3) from the kinetics point of view. This part of the programme has been conducted with both aggregates (the Spratts aggregate is from a different batch) and results are expressed in Table 6. The following comments can be made :

Table 6 : Comparative expansion measurements on concrete prisms, with 2 cement contents

Aggregate	Cement content (kg/m3)		Expansion (%)							
			1 m	2 m	3 m	4 m	6 m	8 m	10 m	12 m
SPRATT	310	1	0.003	0.014	0.034	0.056	0.087	0.109	0.126	0.131
		2	0.005	0.016	0.039	0.069	0.113	0.136	0.159	0.167
		3	0.005	0.018	0.030	0.054	0.095	0.122	0.136	0.145
	410	1	0.009	0.047	0.073	0.093	0.115	0.127	0.137	0.144
		2	0.010	0.047	0.073	0.090	0.112	0.123	0.131	0.136
		3	0.010	0.046	0.076	0.093	0.115	0.128	0.135	0.140
SUDBURY	310		0	0.001	0.003	0.007	0.014	0.027	0.046	0.063
	410		0.001	0.005	0.008	0.018	0.044	0,073	0.094	0.108

1) Whatever the cement content, with the alkali silica reactive Spratts aggregate, the expansion values reached at one year are similar, but expansion goes faster, at earlier ages when the cement content is 410 kg/m3 ; in other words the time elapsed to reach the critical limit of expansion is shorter with the higher cement content.

2) With the slowly expanding sudbury aggregate, the expansion values at one year are higher with the 410 kg/m3 cement content and the critical level of expansion is reached at 6 months, while this requires 10 months with the 310 kg/m3 cement content.

Based on comments 1 and 2, we tentatively propose :

- a 410 kg/m3 cement content should be used instead of the 310 kg/m3 cement content.
- the expansion limit (0.04 % at 1 year) does not need to be changed.
- the time required to identify a reactive aggregate will be around 2 - 3 months in the case of aggregates which expand easily (Spratt's type), and it will decrease from 10 months to approximately 6 months in the case of difficult (slowly expanding) silicate aggregates. This is the most important feature.

6. COMPARATIVE EVALUATION OF RESULTS WITH THE MORTAR BAR METHOD AND THE CONCRETE PRISM METHOD

6.1 Experimental findings

With easily expanding alkali silica reactive aggregates, such as the Spratt and more generally limestone aggregates including some forms of silica, expansion develops faster and reaches higher values, when the aggregate is tested in the the form of mortars : At 12 months, G = 0.33 % with the mortar, while G $\simeq$ 0.15 % with concrete prisms.

For aggregates developing an alkali carbonate reaction (ex: dolomitic Pittsburg aggregate) or late alkali-silicate reaction, results are reversed : the kinetics are faster and expansion values are higher, with concrete prisms. This is somewhat surprising. With the Sudbury aggregate at 12 months G $\simeq$ 0.055 % with mortar bars, and G = 0.108 % with the concrete prisms (cement content = 410 kg/m3).

Although the experimental fact has been established many times and related in papers (see particularly Reference 13), very little has been said to explain such a behaviour. We will approach the problem and tentatively propose explanations.

6.2 Possible Explanations

Can we say firstly, considering the problem from a petrographic and a chemical point of view, that the alkali silicate is something between the alkali-silica and the alkali-carbonate reaction ? Several points suggest this :

The nature of the reactive forms of silica : a mix of rapidly reactive silica (opal, chalcedony...) as in the alkali silica reaction, and a wide variety of silicates, among which are phyllites and clays, which are believed to cause the expansion of coarse aggregate particles in the alkali carbonate reaction.

. The causes of expansion : the alkali silica reaction is associated with the formation of expansive alkali silica gel. In the alkali carbonate reaction, no alkaline gels are formed. In the alkali silicate reaction, the presence of gels is controversial, little or no gels are detected : see, for examples, References 11 and 12.

This means that the reasons why expansion with mortar bar is low in the case of a silicate aggregate are at least partly similar to those encountered with the dolomitic limestone aggregates. This does not mean that alkali silicate reaction equals alkali carbonate reaction.

One possibility is therefore, that, similar to the alkali carbonate reaction, the coarse aggregate expands as a whole, in relation with clay exfoliation. We can also consider that the coarse aggregate expands as a whole, because expansive silica gels (when they are formed) would develop and remain preferentially within the aggregates. But to prepare mortar bars, aggregates are crushed. The fragmentation will break the aggregates preferably in weak parts : porous regions and soft impurities (reactive phases). These, appearing at the new surfaces of the crushed materials, can easily be removed and eliminated, especially when washing the aggregates according to ASTM C227 specifications. This explanation would be in agreement with that given by P.G. Bellew, in a previous publication [14] : He found that expansivity was related to porosity and the percentage of microcrystalline material. He concluded from this study that the mortar bar test could give unreliable results and that expansion of concrete was mainly due to expansion of the individual rocks [15].

We may also think, that in the case of crushed aggregates the gels formed will be localized in small quantities in the vicinity of the aggregates. These small areas of gels would not be strong enough to generate high expansion forces. On the contrary, with coarse aggregates, gels develop in large areas, and the forces generated by expansion are much higher.This question is far from being solved ; it is however a very important one, because, in one way, it approaches the fundamental problem of the mechanisms of reaction.

7. CONCLUSIONS

When using standard tests to estimate the potential alkali-aggregate reactivity, the conditions that they must necessarily fulfil are : reliability and reproducibility. If rapidity can be associated, it is of course interesting but, if not, this quality should be considered as secondary.

Based on this premise, we have decided to investigate the two most popular standards : the ASTM C227 mortar bar test and the CSA A 23.2 - 14 A concrete prism test. Our objectives were first to determine their degree of confidence through a wide experimental programme and then, if they were found acceptable, to use them as basic references to qualify the aggregates of our quarries, and to develop accelerated tests.

From the literature and from our own preliminary results, it was clear that two points were of peculiar importance : the alkali level and the storage conditions. We have therefore concentrated our efforts on these, and our findings and propositions are as follows :

. the alkali level : with both standards, the Na_2O eqv. % should be 1.25 %. This solution, which has already been adopted with the concrete prism test, should be extended to the mortar bar test.

In the case of late or slow expansive reactions, a higher cement content in the concrete composition, which is another way of increasing the alkali content, should be used :

410 kg/m3 seems appropriate.

The storage conditions : special equipment (container and reactor) has been designed to keep the relative humidity (100 %) and the temperature (38°C) constant.

Working with these improved conditions, the results we get with natural reference aggregates (Spratt, Sudbury and Pittsburg) are correct. Results of current studies on French aggregates reinforce our opinion that both these methods can be reliable and reproducible, if correctly used. The concrete prism test has however several points of superiority with regard to the mortar bar method.

It remains that some expansion limits must be revised, and that their response time may be too long in some cases.

REFERENCES

[1] Grattan - Bellew P., Test methods and criteria for evaluating the potential reactivity of aggregates, 279 - 294. 8 th ICAAR, KYOTO, 1989.

[2] Sorrentino D., Ranc R., Cariou B., Methodology of an industrial research laboratory to assess the reactivity of aggregates. Focus on reproducibility problems. 307 - 312, 8 th ICAAR, Kyoto, 1989.

[3] Rogers C.A., Alkali-aggregate reactivity in Canada, 57 - 63, 8 th ICAAR, Kyoto, 1989.

[4] Magni E.R., Rogers C.A., Grattan - Bellew P.E., the influence of the alkali-silicate reaction on structures in the vicinity of Sudbury, Ontario, 17 - 22, 7 th ICAAR, Ottawa, 1986.

[5] Oberholster R.E., Results of an international Inter-Laboratory Test program to determine the potential alkali-reactivity of aggregates by the ASTM C227 mortar prism method, 7 th ICAAR, Ottawa, 1986.

[6] Bérubé M.A., Fournier B., Frenette Y., Evaluation de la performance de divers ciments face aux réactions alcalis granulats. Rapports G GL - 87 - 13 et 15, GRGI, Université Laval, 1987.

[7] Rogers C.A., Hooton R.D., leaching of alkalies in alkali - aggregate reaction testing. 327-332, 8 th ICAAR, Kyoto, 1989.

[8] Hooton R.D., Rogers C.A., Evaluation of rapid test methods for detecting alkali-reactive aggregates, 439 - 444, 8 th ICAAR, Kyoto, 1989.

[9] Ranc R., Sorrentino D., Cariou B., Réactions silico-alcalines. Mise au point de méthodes fiables. Annales ITBTP, n° 480, 109 - 135, 1990.

[10] Bérubé M.A., Fournier B & Frenette J. : Evaluation du potentiel de réactivité alcaline des granulats produits par trois carrières appartenant à DEMIX Inc. et situées respectivement à Laval (zone nord et sud), Saint-Jacques-le-Mineur et Varennes. Rapport GGL-88-09, Groupe de Recherche en Géologie de l'Ingénieur, Université

Laval, 107p. 1987.

[11]Alkali-aggregate reaction. CSA CAN 3-A23.1 - M77 Appendix B, CanadianStandards Association Supplément n° 2, p.3, 1986.

[12]Bérubé M.A., and Coll. Les réactions alcalis granulats dans le béton. Cahier 1, cours GGL-AO487, p. 24, Université Laval, mai 1988.

[13]Grattan - Bellew P.E., Re-evaluation of standard mortar and concrete prism tests for alkali-aggregate reactivity, vol 16, n° 94, 243 - 250, Matériaux et constructions, 1983.

[14]Grattan - Bellew P.E., Study of expansivity of a suite of quartzwakes, argillites and quartz arenites. Proceedings of the 4 th Int. Conf. on the effects of alkalies in cement and concrete, 113-140, Pub. CE-MAT-1-78, Purdue University (1978)

[15] Rogers C.A. Alkali reactions in Ontario P. 5, in Concrete Alkali-Aggregate Reactions. 7 th ICAAR, Ottawa, 1986.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

THE MICROBAR METHOD, AN ACCELERATED EXPANSION TEST FOR EVALUATING AGGREGATES. ASSESSMENT OF CANADIAN AGGREGATES.

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ABSTRACT

The alkali reactivity of 8 Canadian and 9 foreign aggregates was evaluated using a modified version of the Chinese autoclave method developed by Tang et al. The major changes are described; the various aspects of reproducibility were thoroughly assessed and quantified as well as the influence of the granulometric distribution.

The diagnosis delivered in 3 days by this microbar method is compared in 13 cases with field records and, by defect, in 4 cases, with other evaluation methods (ASTM C227, NBRI, ACNOR..). Provisional reactivity thresholds are proposed. The field soundness or reactivity is found to be always correctly predicted, even in the case of ASTM C227 failure, with the only exception of alkali-carbonate reactive aggregates. The major advantages of this approach are outlined.

1. INTRODUCTION

The need for a rapid method for evaluating the alkali-reactivity of aggregates has long been recognized. Such a method, in addition to its rapidity, must be reliable, reproducible and applicable to both alkali-silica and alkali-silicate reactive aggregates. Finally it should ideally also feature easy handling.

The method proposed here, thereafter referred to as the MICROBAR method, is largely derived from the autoclave Chinese test of Tang et al [1,2]. This is the quickest of

the accelerated mortar bar expansion methods which have been developed so far [3]. In our search for an accelerated expansion test, we have selected this approach because of some unique advantages compared to other accelerated methods namely (1) simplified time-saving preparation of the aggregate ; (2) easy specimen treatment and space-saving storage; (3) bench size lab equipment.

The method was improved by designing a specific equipment (moulds, studs..), defining more closely some steps , when needed, and testing the aggregate at three cement/aggregate ratios instead of one as originally proposed [1,2].

The MICROBAR method is now routinely performed at the Ciments Français Group and more than 60 natural aggregates have been evaluated. We report here the results obtained with Canadian and some US and foreign aggregates well known for either their reactivity or their good field performance.

2. EXPERIMENTAL METHODS

2.1 Materials

2.1.1 Aggregate The aggregate is crushed and ground until it all passes 630 µm . The reduced fraction is sieved and washed and the portion passing 150 µm is discarded. The fraction 150-630 µm, which is used for testing, was adapted from the original 150-750 µm [1,2] to the French AFNOR sieve standard . This granulometric range is reported to give the highest expansions with the conventional ASTM C227 method [3,4,5].

More than 60 reactive and sound natural aggregates from various types and origins have been tested so far. The results presented in this paper concern a selection of 8 Canadian, 3 US, 3 Italian, 2 Danish and 1 South-African aggregates. Their names together with their mineralogical type are given in tables 1 and 2. A few reference and synthetic materials were also used, namely the Chapteuil opal from the French Massif Central, a cristobalite sample kindly supplied by Blue Circle Industries [6] and fused silica manufactured by Tilcon Ltd (UK).

2.1.2 Cement Two type III Ordinary Portland cements (OPC) from the Ciments Français Cantin and Barlin plants were used, containing respectively 0.49 and 1.03 % Na_2O equivalent. As shown in table 3, their C_3A contents are also markedly different.

Table 1 : MICROBAR expansions and standard deviations (I) at c/a = 2, 5, 10 for a selection of aggregates.Comparison with field records or previous testings
R : reactive S : sound P : potentially reactive

Aggregate	Expansion I			Reactivity		
	2	5	10	Field	ASTM 227	Other
U.S.A :						
1. KI dolomite	0.030 ± 0.003	0.016 ± 0.001	0.011 ± 0.003	S		
2. CF limestone	0.037 ± 0.008	0.015 ± 0.001	0.011 ± 0.002	S		
3. Ottawa quartz	0.029 ± 0.003	0.023 ± 0.003	0.016 ± 0.005		S	
South Africa :						
4. Hornfels						
0.6 - 0.18mm	0.140 ± 0.010	0.092 ± 0.006	0.069 ± 0.005)			)
0.3 - 0.6 mm	0.189 ± 0.012	0.149 ± 0.009	0.096 ± 0.008)R			)R(NBRI)
0.15 - 0.3mm	0.366 ± 0.013	0.179 ± 0.004	0.116 ± 0.003)			)
Italy :						
5. Roma limestone	0.004 ± 0.005	0.024 ± 0.009	0.024 ± 0.010		S	S(NBRI)
6. Quartz IC	0.216 ± 0.004	0.289 ± 0.004	0.263 ± 0.004	R		
7. T limestone	0.197 ± 0.012	0.132 ± 0.012	0.064 ± 0.006		R	R(NBRI)
Denmark :						
8. Nymölle sand	0.184 ± 0.007	0.107 ± 0.011	0.072 ± 0.006	R	R	R (MBT)
9. Kallerûp sand	0.129 ± 0.013	0.088 ± 0.012	0.069 ± 0.002	R	R	R (MBT)

1 : Eastern Industries 2 : Coplay Frederick

Table 2 : MICROBAR and standard deviation (I) at c/a = 2, 5, 10 for a selection of Canadian aggregates. Comparison with field records or previous testings
R : reactive S : sound P : potentially reactive

Aggregate	Expansion I			Reactivity		
	2	5	10	Field	ASTM C 227	Other
10. Nelson limestone	0.021 ± 0.001	0.012 ± 0.005	0.006 ± 0.001	S		
11. Spratt limestone	0.307 ± 0.025	0.202 ± 0.013	0.102 ± 0.010	R	R	R (NBRI, concrete)
12. Sudbury greywacke, argillite, sandstone gravel	0.185 ± 0.006	0.077 ± 0.008	0.043 ± 0.003	R	S	R (concrete)
13. Beauharnois sandstone	0.129 ± 0.005	0.058 ± 0.009	0.047 ± 0.010	R	S	R (concrete)
14. Pittsburg dolomite	0.043 ± 0,006	0.016 ± 0.003	0.009 ± 0.008	R*	S	R* (concrete)
15. Beauceville rhyolitic tuf	0.197 ± 0.004	0.136 ± 0.007	0.101 ± 0.010	R		
16. CBM quartz sand	0.019 ± 0.004	0.012 ± 0.002	-0.009 ± 0.003	S	R	S (NBRI)
17. Springhill NB greywacke	0.084 ± 0.004	0.077 ± 0.004	0.059 ± 0.003	R	S	

* Alkali carbonate reactive

Table 3: Alkali content and composition of the cements used for the MICROBAR tests

		Cantin	Barlin
Na_2O	%	0.29	0.23
K_2O	%	0.30	1.22
Na_2O_{eq}	%	0.49	1.03
SO_3	%	2.06	3.03
MgO	%	0.69	1.05
C_3S	%	66.4	49.2
C_2S	%	11.5	19.5
C_3A	%	3.0	13.1
C_4AF	%	11.1	7.7

2.2 Apparatus

An equipment was designed for mixing small amounts of mortar ($\sim$150 g); it can be retrofitted in a few minutes on a conventional AFNOR standard mixer . Moulds made of a fluoropolymer were also designed with special studs for casting 4 1*1*4 cm mortar bars.

The length measurements are carried out at 20°C with an incremental Heindenhain meter and are accurate to ± 2 µm. Vapour curing is achieved by suspending the microbars in a covered basket above boiling water inside a Pyrex flask fitted with a condenser. A simple oven at 150 °C and individual cylindrical stainless steel containers that can accomodate 4 bars are used for the alkaline cure. Details of the complete equipment are available upon request.

2.3 Procedure

For each aggregate three tests are run, corresponding to cement/aggregate ratios (c/a) respectively equal to 2, 5, 10. Tang [1] suggested a single mix with a cement/aggregate ratio of 10, but the potential reactivity of aggregates is better assessed with the additional tests, as it will be shown later.

The water/cement ratio is equal to 0.3, and the mix water is enriched with a NaOH solution in order to reach an equivalent Na_2O content of 1.5% by weight of cement. Four mortar bars of size 1*1*4 cm are prepared for each test. After curing for 24 hours in a 100% RH chamber at 20 °C, the mortar bars are demoulded and their initial length measured. The micro-bars are then submitted to a water vapor curing at 100 °C during 4 hours, cooled down to and stored at room temperature overnight. The alkaline curing is carried out the next day: the bars are soaked in a 10% KOH solution and

kept for 6 hours at 150 °C. Finally they are cooled down to room temperature and their final length measured.

The length change is calculated for each microbar and the expansion result, expressed in % of the initial length, is the average length change on the four microbars of a single test. For any aggregate, the highest of the expansions obtained with the three different cement to aggregate ratios is retained for classifying the aggregate.

3. RESULTS AND DISCUSSION

3.1 Reproducibility

The reproducibility of the method was assessed at three levels: (1) standard deviation for the 4 bars of a single mix , (2) standard deviation for different mixes run on the same composition, and (3) variations due to the operator(s).

3.1.1 Per mix When the four bars of the same mix are considered, the expansion difference of any specimen from the average value is generally better than 10 % for expansions in the range of 0.1% and above. Examples are given thereafter in table 4 for three Canadian aggregates. The precision is thus considered satisfactory.

Table 4: Reproducibility by mix for 3 Canadian aggregates using Cantin cement. Expansion is expressed in %.

		Sudbury	Nelson	Beauharnois
c/a=2	I	0.183	0.020	0.126
	II	0.177	0.020	0.126
	III	0.189	0.022	0.129
	IV	0.189	0.020	0.136
average		0.185±0.006	0.020±0.002	0.129±0.004
c/a=5	I	0.087	0.015	0.067
	II	0.079	0.017	0.057
	III	0.069	0.005	0.062
	IV	0.072	0.012	0.047
average		0.077±0.008	0.012±0.006	0.058±0.005
c/a=10	I	0.039	0.005	0.022
	II	0.042	0.007	0.039
	III	0.044	0.005	0.044
	IV	0.046	0.005	0.042
average		0.042±0.003	0.006±0.001	0.037±0.008

3.1.2 Per aggregate The accuracy of the method was checked by repeating the test several times on some aggregates. The results are given in table 5 for three different aggregates.

Table 5: Expansion and standard deviation σ (expressed in %) for repeated tests; c/a=10; Cantin cement.

run No	Spratt	AFNOR sand	Ottawa quartz
1	0.085± 0.008	0.051± 0.004	0.017 ± 0.003
2	0.113± 0.002	0.067± 0.002	0.020 ± 0.005
3	0.097± 0.008	0.053± 0.012	0.019 ± 0.005
4	0.108± 0.014	0.065± 0.002	0.013 ± 0.004
5	0.094± 0.006		
6	0.096± 0.002		
7	0.098± 0.011		
mean	0.099± 0.009	0.059+±0.008	0.017 ± 0.003

It is readily observed that the values obtained for the individual runs generally lies between ± σ of the mean value for expansions in the range of 0.1% and above. With the only exception of run No2 on the Spratt aggregate, all values lie within ± 2 σ of the mean value, which gives confidence in the results.

3.1.3 Influence of the operator One trained and two occasional operators ran a series of tests on 5 aggregates of different AS reactivity (table 6).

Table 6: Expansions and standard deviations σ (%) obtained for 5 aggregates by three different operators. 1:trained operator; 2 and 3: occasional operators; Cantin cement; c/a=10.

aggregate	1	2	3	mean
Ottawa quartz	.019± .005	.025± .012	.005± .015	.016
Porphyry	.084± .006	.069± .005	.074± .005	.076
Brix sandstone	.080± .005	not completed	.074± .005	.077
Spratt	.102± .012	.097± .011	.095± .011	.098
Chapteuil opal	.965± .070	.890± .040	.940± .050	.932

The mean value always lies in the ± 2 σ range of the individual expansion results. The evaluation of a given aggregate can be therefore considered independant of the

human factor. Further investigations have indicated that one critical point for the reproducibility at the early step of the procedure is the weighing of the materials.

3.2 Influence of the cement

As the composition of the cement used might have been suspected to affect expansion, the tests on a large number of aggregates were systematically duplicated using Cantin and Barlin OPCs and a cement to aggregate ratio of 10. It is reminded that the alkali content is systematically adjusted to 1.5% by weight of cement.

The results are illustrated in figure 1 where the expansions obtained on 25 aggregates with Barlin cement are plotted versus those observed with Cantin cement. With the exception of two cherts, all the points are located around the line of slope equal to 1. Thus the expansion results are identical, within the experimental precision, with both cements and therefore independant of the cement used.

We therefore conclude that the MICROBAR method evaluates specifically the expansion due to AS reactivity of the aggregates and is not affected by other parameters.

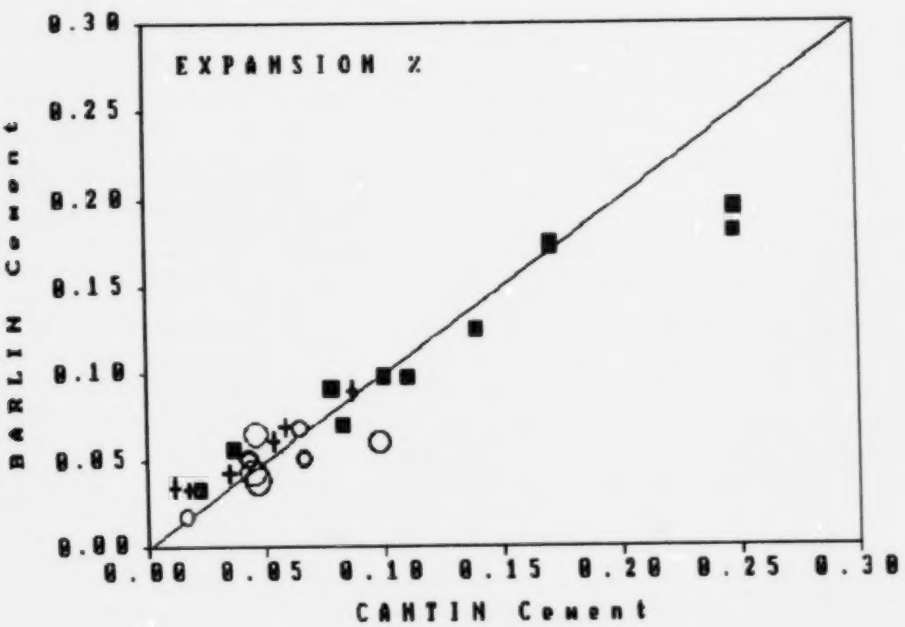

Figure 1: Influence of the cement. ■:gravel O:sand +:limestone.

3.3 Influence of the concentration of reactive silica

The expansion caused by increasing amounts of highly reactive aggregates (opal, cristobalite and fused silica) in substitution with a non reactive aggregate (Ottawa quartz) was measured, using a cement to aggregate ratio equal to 10 and Cantin cement. Because these materials are almost exclusively composed of silica (SiO_2 >99%), this sheds some light on the effect of the crystallographic form and concentration of reactive silica.

Only for fused silica is the expansion proportional to the percentage of reactive component from 0 to 100% substitution (Figure 2). For opal, the curve levels off at about 80% whereas cristobalite displays an intermediate behavior. This typical feature is reminiscent of the pessimum effect, which has been extensively described for opals [6], but should be considered with caution as the final curing takes place in a 10% KOH solution and thus in a potentially drifting SiO_2/Na_2O equ.ratio.

In the conditions of this experiment , i.e. cement /aggregate = 10 , the lowest content of opal which can be accurately detected in an aggregate is about 5%. When the cement to aggregate ratio is decreased to 5 or 2, lower amounts of opal could be evidenced (about 2%).

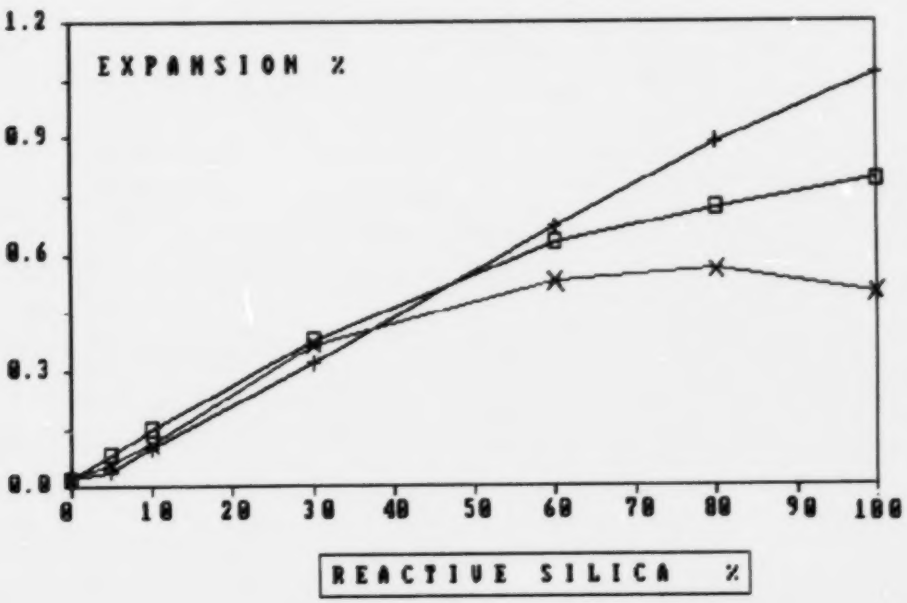

Figure 2: Influence of the type and amount of reactive aggregate in substitution of sound quartz. +:fused silica □:cristobalite; x:opal.

3.4 Influence of the granulometry

The effect of the particle size of the reactive aggregate was evaluated using Cantin cement and the Hornfels aggregate well known for its reactivity [7]. The results are given in Table 1.

The results show a clear dependance of the expansion magnitude on the granulometric range, the 150-300 µm fraction leading to higher expansions than the coarser fractions; this effect has already been documented in the literature [3,4,5]. In relation with the MICROBAR method, the point that must be stressed is that the 150-630 µm fraction used is indeed the most sensitive. Coarser fractions, as used in the ASTM C227, are not critical for the evaluation of the intrinsic reactivity of the aggregate tested. In addition, and somewhat paradoxically, these data tend to support the idea that a close control of the granulometric distribution in the 150-630 µm range is not necessary, therefore comforting the simplified preparation of the aggregate proposed by Tang et al with respect to the ASTM C227.

3.5 Evaluation of aggregates

Tables 1 and 2 summarize the expansion results obtained for a selection of 17 aggregates, together with a brief description and past field performance or other available information.

3.5.1 Variation of expansion versus cement to aggregate ratio Most of the samples display an increasing expansion with decreasing cement/aggregate ratio. This trend is of particular interest for slowly expanding aggregates (Sudbury, Beauharnois) which otherwise do not reveal their reactivity at ratios of 5 and 10. A few samples (quartz and gravels) on the contrary are characterized by a reverse trend (see figure 3). This is thought to be due to the different nature of the reacting silica , but further studies are needed.

For this reason a determination at a single cement/aggregate ratio cannot reliably predict the reactivity of an aggregate. Another advantage of this triplicate procedure is that aggregates having different reactivities can be differentiated more clearly.

3.5.2 Provisional threshold Tang et al have proposed an expansion of 0.11% as a limit for the reactivity. Taking into account the results exposed in table 1 and a number of results on other sound and reactive aggregates, we tentatively and provisionally suggest the following limits for the MICROBAR method :

expansion < 0.08 % sound aggregate
0.08<expansion<0.1 % potentially reactive aggregate

expansion > 0.1 % reactive aggregate

where the MICROBAR expansion is taken as the highest value obtained with the three cement to aggregate ratios. These limits should be confirmed by a round robin validation campaign.

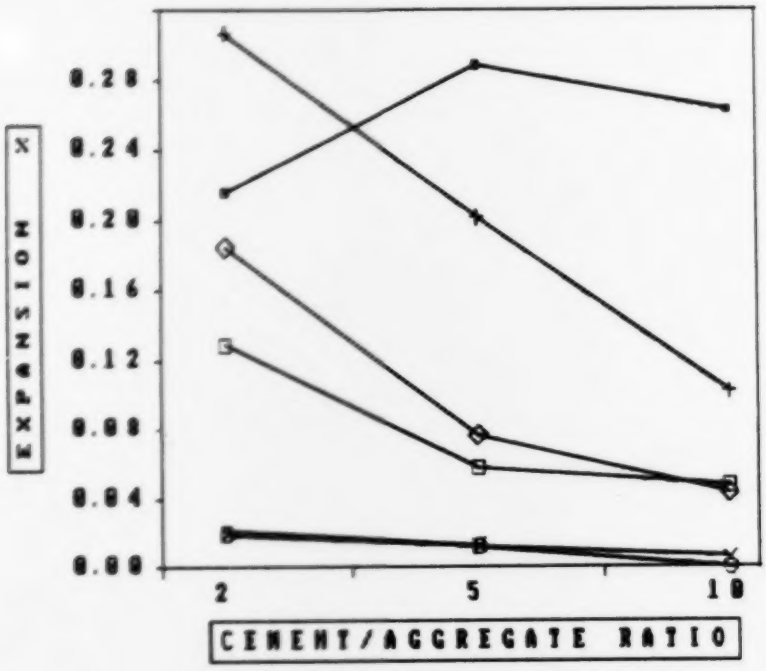

Figure 3: MICROBAR expansions versus c/a. +:Spratt; ▪:Quartz IC; ◇:Sudbury; □:Beauharnois; x:Nelson; O: CBM sand.

3.5.3 Comparison with other expansion tests The methods used for testing the materials of tables 1 and 2 are of very different nature (ASTM C227, ACNOR, NBRI, MBT..)[3,7,8,9,10,11,12]. Consequently, it appears more consistent to establish a "one bit" type of correlation (Figure 4) when comparing the different methods rather than trying to plot expansion percentages versus one another, at least at this stage.

The diagnoses given by the MICROBAR test are in close agreement with the available information. The well-known sound aggregates show very low expansions, while rapid or slowly expanding materials are unequivocally declared

reactive. Very few aggregates fall into the intermediate class.

As previously stated by Tang [1,2] the test evaluates the alkali-silica and alkali-silicate reactivity of aggregates. It is not applicable for alkali-carbonate reactivity, and this is illustrated by the Pittsburg limestone, which yields a low expansion whatever the cement to aggregate ratio (Table 2).

3.3.4 Canadian aggregates The sound CBM quartz sand and Nelson limestone give maximum expansions of 0.02%, in agreement with former findings. The Sudbury gravel and Beauharnois sandstone, which have a well established field record of reactivity but do not produce expansions with the mortar bar ASTM C227 test [9,11], are classified reactive with the MICROBAR test. The reactive and well known Spratt [9] and Beauceville [12] aggregates also produce significant expansions. Springhill greywacke, a slowly reactive aggregate, is one of the very few examples falling in the grey zone of potential reactivity proposed in 3.5.2. The Canadian aggregates tested so far all display a decreasing expansion with decreasing c/a ratio (Figure 3).

The alkali-carbonate reactivity of the Pittsburg aggregate is not unveiled with this quick mortar bar method, as discussed above.

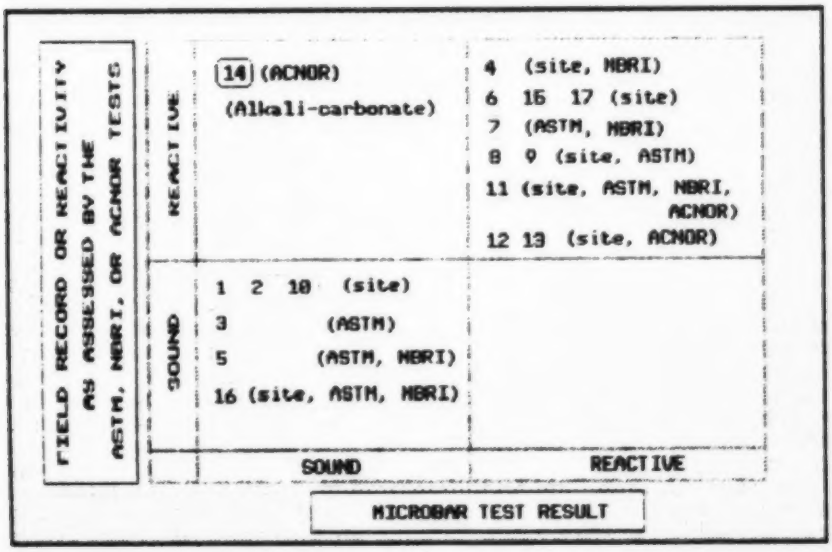

Figure 4:"One bit" correlation between the verdict given by the MICROBAR method on the one hand and field record or other tests in the other hand.

4. CONCLUSIONS

4.1 Field of application

The MICROBAR method provides a reliable evaluation of aggregates with regard to their alkali-silica and alkali-silicate reactivity. It cannot be used for assessing the alkali-carbonate reactivity.

4.2 Advantages

The method is rapid and does not require tedious preparation work nor a large amount of aggregate. Hence, it allows to carry out more tests than the cumbersome traditional tests. This is of particular interest to assess a source of aggregate in great detail, for example in the case of heterogeneous quarries. Certain sandstones, for example, show distinctive veins of reactive rock which can only be properly evaluated by multiplying the tests. The authors have also tested limestone samples from a single source but with a wide range of reactivity.

The reliability of the method has been improved by performing the test at three cement/aggregate ratios. Slowly expanding materials are better detected at lower c/a ratios.

REFERENCES

[1] Tang M.S., Han S.F. and Zhen S.I., A rapid method for identification of alkali-reactivity of aggregates, Cement and Concrete Research, 13, 417,1983.

[2] Tang M.S., Han S.F., Yuan M.Q., Ye Y.F. and Lu Y.N., Applications of autoclave rapid test method in practical engineering projects in China, Proceedings of the 7th International Conference on Alkali-Aggregate Reactions, Ottawa, 1986, Grattan-Bellew ed. (1987).

[3] Grattan Bellew P.E., Test methods and criteria for evaluating the potential reactivity of aggregates, p 279-294, Proceedings of the 8th International Conference on Alkali-Aggregate Reaction, Kyoto (1989).

[4] Kawamura M., Takemoto K. and Hasaba S., Application of quantitative EDAX analysis and micro-hardness measurements to the study of Alkali-Silica Reaction mechanisms, p 167-174, Proceedings of the 6th Int. Conference on Alkalis in Concrete, G.M.Idorn and S.Rostam eds, Copenhagen (1983).

[5] Hobbs,D.W., Alkali-Silica Reaction in Concrete, Thomas Telford Ltd.,London (1988).

[6] Lumley J.S., Synthetic cristobalite as a reference reactive aggregate, Proceedings of the 8th Int. Conference on Alkali-Aggregate Reaction, Kyoto, K.Okada, S. Nishibayashi and M. Kawamura eds (1989).

[7] Oberholster R.E., Alkali reactivity of siliceous rock aggregates: diagnosis of the reaction, testing of cement and aggregate and prescription of preventive measures, p419-433, Proceedings of the 6th International Conference on Alkalis in Concrete, G.M. Idorn and S.Rostam eds, Copenhagen (1983).

[8] Thaulow N. and Olafsson H., Alkali-silica reactivity of sands, comparison of various test methods, Nordtest Project, p 359-364, Proceedings of the 6th International Conference on Alkalis in Concrete, G.M. Idorn and S.Rostam eds, Copenhagen (1983).

[9] Rogers C.R., General information on standard alkali-reactive aggregates from Ontario, Canada, 1st edition, Ministry of Transportation.

[10] Magni E.R., Rogers C.A. and Grattan-Bellew P.E., The influence of the alkali-silicate reaction on structures in the vicinity of Sudbury, Ontario, p 17-22, in Concrete Alkali-Aggregate Reactions, P.Grattan-Bellew ed, Noyes Publication, 1987.

[11] Albert P. and Raphael S.,Alkali-reactivity in the Beauharnois powerhouses, Beauharnois, p 10-16, in Concrete Alkali-Aggregate Reactions, P.Grattan-Bellew ed, Noyes Publication (1987).

[12] Bérube M.A, personal communication.

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COMBATING THE ALKALI-REACTIVE AGGREGATE PROBLEM IN THE SUDBURY AREA - A GEOLOGICAL APPROACH

by

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ABSTRACT

An aggregate resources inventory project was undertaken to help alleviate an alkali-aggregate reactivity problem recognized in the Sudbury area in northern Ontario. The concentration of alkali-reactive gravel within glacial deposits near Sudbury is a result of geologic processes active during the last major continental glaciation in North America. By combining an understanding of these processes and a knowledge of bedrock geology with an extensive sampling program, several deposits were identified that meet Ontario Ministry of Transportation (MTO) specifications for content of alkali-reactive gravel.

INTRODUCTION

Argillites, greywackes and sandstones of the Precambrian Huronian Supergroup are major rock types which form the bedrock in the vicinity of Sudbury, northern Ontario (Figure 1 [1, 2]). This situation leads to the occurrence of these rocks as the dominant lithologies in the glacially derived sands and gravels in the same area.

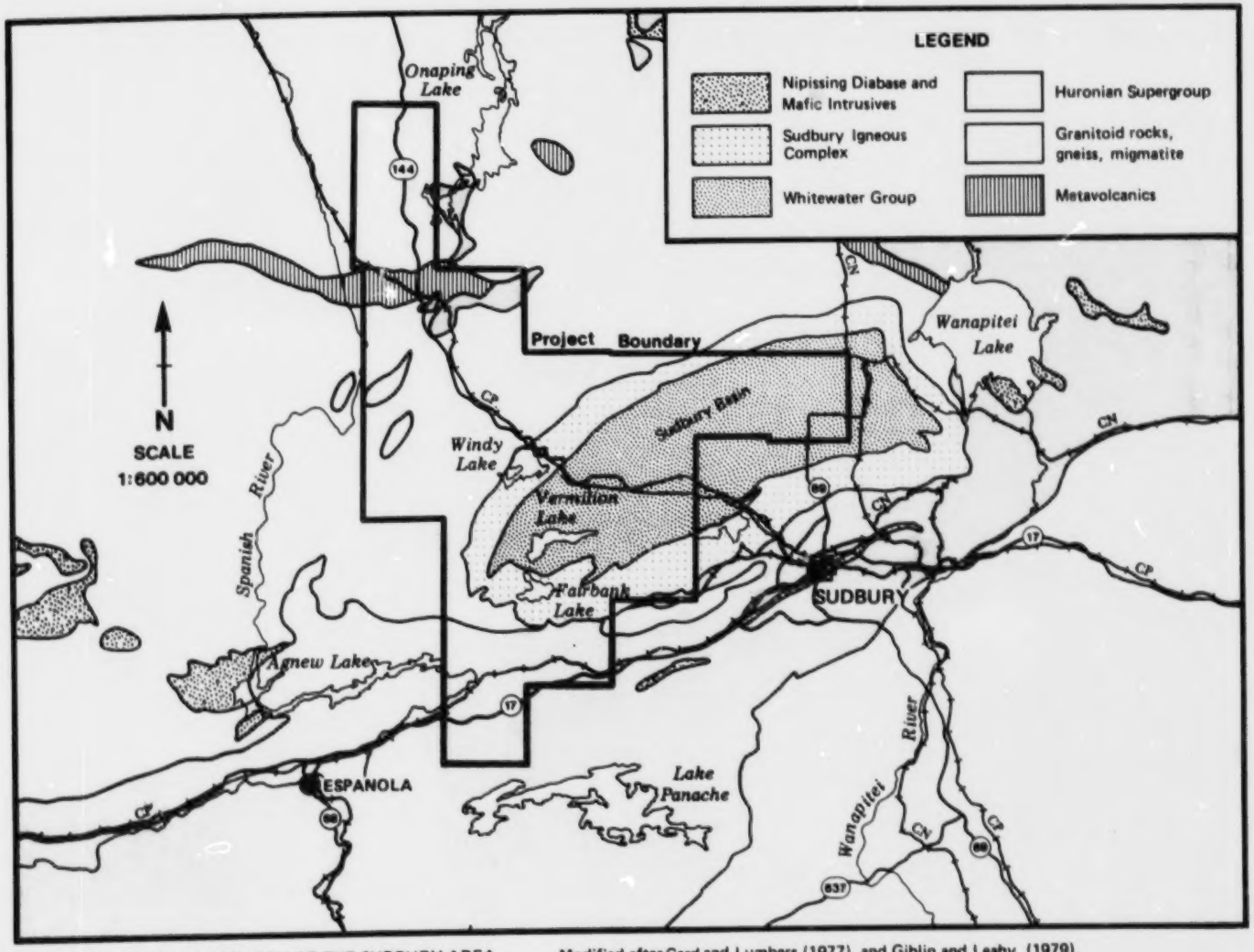

FIGURE 1: BEDROCK GEOLOGY OF THE SUDBURY AREA Modified after Card and Lumbers (1977), and Giblin and Leahy (1979).

The evaluation of in-situ performance and the conduct of concrete expansion tests has shown that these rock types are generally quite reactive with the alkali component of Portland cement [3, 4, 5, 6, 7, 8]. Concrete structures affected by the reaction in the Sudbury area include bridges, dams, retaining walls, sidewalks, curbs and gutters which are subject to a moist environment and/or road salts. Because of this reaction, MTO has limited to 15 percent, the maximum amount of alkali-reactive gravel in concrete aggregate in the Sudbury area unless testing indicates that the materials will provide satisfactory performance. As the major aggregate sources in the area were known to contain between 65 and 90 percent alkali-reactive gravel [5], aggregate required for high service demand concrete had to be hauled in from the North Bay area (approximately 80 km to the east) or quarried locally. Such a haulage distance added considerably to the delivered cost of good quality aggregate in the Sudbury area. The costs associated with the alternative of quarrying hard, durable and non-reactive types of bedrock within the Sudbury area were comparable to the high haulage costs. Thus a project [9] was initiated to see if deposits containing low amounts of alkali-reactive aggregate could be identified in the Sudbury area. This project is part of the ongoing program of the Ontario Geological Survey to prepare Aggregate Resources Inventory Reports.

Various geologic processes were responsible for the concentration of Huronian derived particles within gravel deposits in the area. Based on knowledge of the distribution of Huronian bedrock and the direction of glacial ice movement, interest was focussed on an area west of Sudbury for field investigations. Aggregate deposits within this area were expected to have the lowest amounts of alkali-reactive material within a reasonable trucking distance of the City of Sudbury.

FIELD AND LABORATORY STUDIES

Methods used to assess the aggregate resources west of Sudbury initially involved the compilation of all available data including bedrock and surficial geology maps and reports, as well as pit and aggregate quality information on file with MTO. Aerial photographs were also examined to identify potential deposits. These deposits were subsequently examined in the field. Field methods involved the examination of both natural and man-made exposures of granular material. Observations made at these exposures included the estimation of the percentage of various grain sizes, and the amount of objectionable material present.

Sedimentary features were also identified to help determine the origin of each deposit. Backhoe excavation, soil probing and geophysical techniques were used to assess subsurface material in areas of limited exposure.

Field investigations also involved an extensive sampling program. Representative samples of aggregate weighing 15 to 45 kg were taken from exposures and test pits. Preliminary examinations were conducted in the field on 83 samples to roughly estimate the percentage of reactive Huronian particles within the gravel fraction of the aggregate. The results of these field examinations allowed for the rapid identification of high potential areas requiring further detailed study. After completion of field work, over 70 samples were submitted to the MTO laboratories. These samples were analyzed for grain size distribution and four aggregate quality tests were performed. These tests include the Los Angeles abrasion and impact test, absorption and the Magnesium Sulphate soundness test and petrographic analysis. Petrographic examination provided Petrographic Numbers for the various samples and the percentage by weight of alkali-reactive particles within the gravel fraction of the aggregate.

GLACIAL GEOLOGY

A study of the glacial history of the area was an integral part of the investigation. During the last major advance of the continental ice sheets during the Late Wisconsinan, glacial ice advanced in a south to southwesterly direction across the area [10, 11, 12]. The advancing ice eroded the Precambrian basement rock which it transported as rock debris, the debris thus reflecting the locally overridden bedrock. As the Huronian Supergroup underlies a large part of the area, especially northeast of Sudbury, the Supergroup rocks were a major source of debris. Transportation of rock debris at the base of the advancing glacier scratched bedrock surfaces forming striae oriented in a general 180° to 200° direction with local divergences of 210° to 240° within and south of the Sudbury basin [12]. Thus the direction of movement of the glacial ice was established.

The rock debris was either deposited directly by the glacial ice as till or carried by glacial meltwaters and/or wind. A discontinuous layer of the till was deposited throughout the project area by the advancing ice. This sandy till generally exists as a thin veneer over bedrock.

Till is usually not well suited for aggregate use as it often contains excess fines and abundant oversize material.

The melting back of the glacial ice front from the Sudbury area occurred approximately 10,000 to 12,000 years BP [13, 14, 15]. Large volumes of sediment-laden water were released from the ice sheet as it melted. These meltwaters laid down aggregate-bearing glaciofluvial deposits within the project area. During a temporary halt in the position of the retreating ice front, an east-trending moraine ridge was also deposited along the rim of the Sudbury Basin. This moraine consists of a mixture of till, sand, gravel and boulders.

Glaciofluvial deposits which were closely associated with the ice during deposition are known as ice-contact deposits. Ice-contact features typically consist of irregularly-bedded, poorly-sorted, sand and gravel. Eskers are one variety of ice-contact deposit common in the area. Eskers form segmented, sinuous ridges and trend in a general north to northeast direction. They were deposited by meltwaters flowing in tunnels under the ice or in reentrants at the ice front. Hummocky deposits of undifferentiated ice-contact stratified drift are also abundant in the area. The origin of these complex features can be attributed to a variety of depositional environments and many of these deposits are associated with esker systems.

Another variety of glaciofluvial deposit in the study area is outwash. Outwash was deposited by meltwaters flowing in bedrock depressions beyond the ice margin. The outwash primarily consists of well-stratified and uniformly-bedded sand and gravel and is the most widespread aggregate deposit type in the area.

As the ice front melted back to a position north of the Sudbury Basin, the Basin, along with low-lying areas in the southern part of the project area, was inundated by postglacial lake waters. Where glaciofluvial systems entered the lake along the rim of the Basin, broad glaciolacustrine deltas were formed. The deltas consist largely of well-stratified and uniformly-bedded sand and gravel which is well suited for aggregate extraction. Finer materials were deposited on the floor of the glacial lake. Glaciolacustrine sediments consisting of silty fine sand, silt and clay are the predominant surficial material exposed in the Basin.

Erosional and depositional activities have been minimal since the disappearance of the ice sheets and the lowering of lake waters to present-day levels.

AGGREGATE QUALITY AND SUITABILITY

Results of laboratory testing indicate that the aggregate in most of the study area is hard and durable, and is of sufficient quality for uses including hot-laid asphaltic concrete, Granular A and B Type I, and surface treatment aggregates. The results of the four quality tests for the most part fall within MTO specifications for these products. The exception to this statement is the gravel found in an area to the southwest of the Sudbury Basin. The problem in this area is not the presence of Huronian Supergroup rocks but rather the presence of soft shale (argillite) and volcanic particles, fissile slates and limonite-coated particles, all of which originated from the bedrock of the Whitewater Group which forms the central part of the Sudbury structure.

The deposits containing aggregate suitable for high service demand concrete are restricted to three areas, 23 km or more from the Sudbury market (Figure 2). The best potential sources in these areas are large deltaic features deposited along the northeastern rim of the Sudbury Basin and contain between 2 to 14 percent alkali-reactive gravel. These low percentages of alkali-reactive material not only satisfy the MTO specifications but are in stark contrast to the more than 65 percent alkali-reactive content of gravel from the major suppliers of the Sudbury market (15 to 22 km northeast of the city).

Various factors may be responsible for variations in the concentrations of alkali-reactive aggregate within the project area. They include: the interrelationships between ice-flow direction; the distribution of Huronian bedrock and glacio-dynamics along with the distance of transport; and the environment of deposition of the sediment. Because of the often complex relationships between these factors, it is difficult to predict the amount of alkali-reactive aggregate that will be found in an area. However, some generalizations can be made which may aid future exploration for concrete aggregate.

Deposits that are located in close proximity to Huronian bedrock and in a down-ice position often contain high concentrations of alkali-reactive particles. Such is

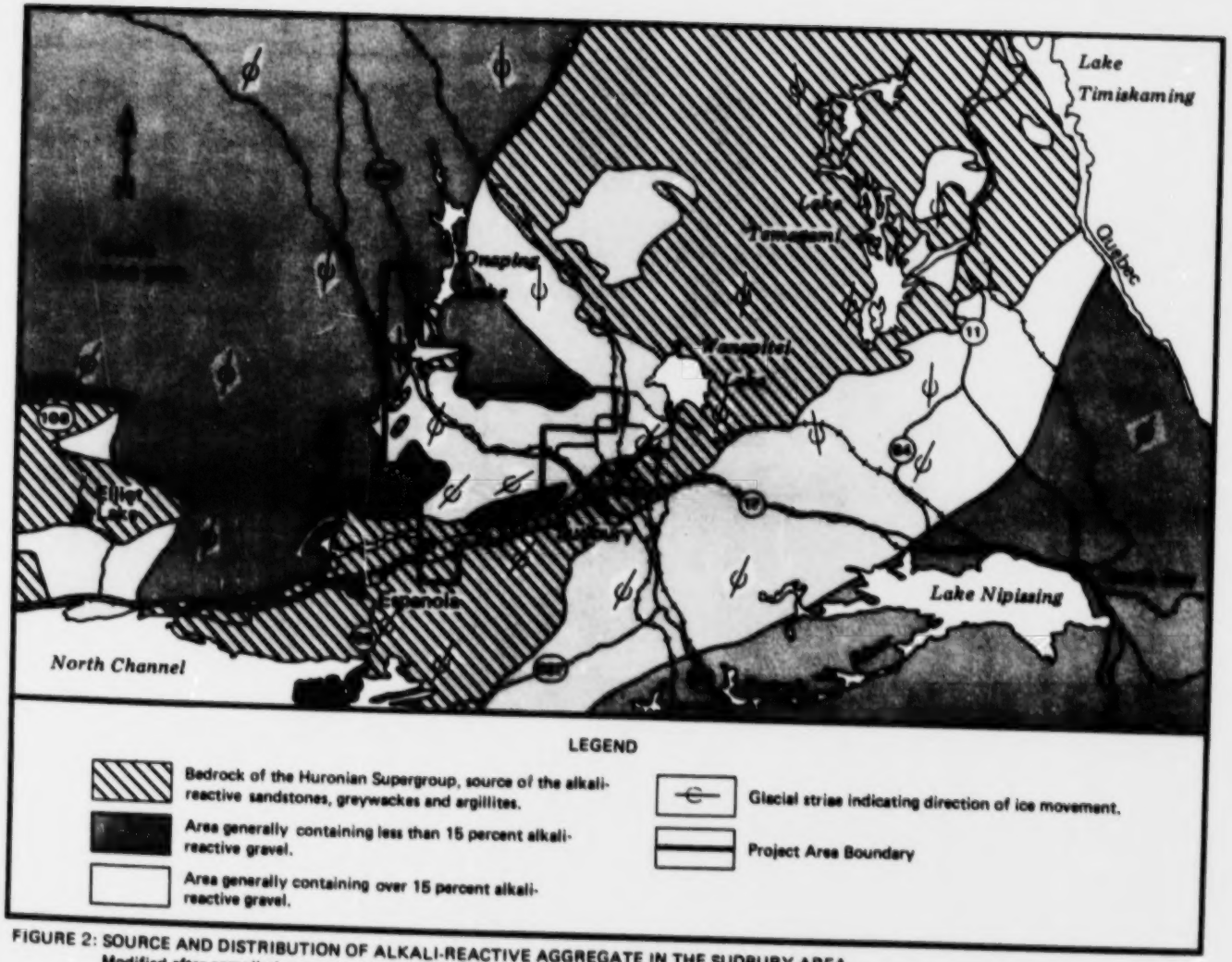

FIGURE 2: SOURCE AND DISTRIBUTION OF ALKALI-REACTIVE AGGREGATE IN THE SUDBURY AREA
Modified after compilation map by Northern Region of M.T.O. based on petrographic numbers of samples from various pits.

the case with deposits located in the southern and eastern corners of the study area which contain over 45 percent reactive gravel. Conversely, the deposits located in the northwestern corner of the area contain only low amounts of alkali-reactive gravel as they are not situated directly down-ice from Huronian bedrock. Deposits that are located down-ice from an area containing a local but irregular distribution of Huronian bedrock, as found in the west-central part of the project area, often exhibit wide variations in the concentrations of alkali-reactive aggregate they contain.

In general, higher concentrations of Huronian particles are evident in the fine gravel component of the coarse aggregate. This relationship was observed in many samples taken during field investigation. Therefore, crushing of oversize material with a primary crusher may increase the suitability of the aggregate for concrete use. In certain areas, however, higher concentrations of reactive gravel may occur in the coarse gravel fraction of the aggregate. This relationship may be expected to occur in deposits situated in close proximity to Huronian bedrock. The sand fraction of the aggregate in the Sudbury area is not considered to be deleteriously alkali-reactive.

Deposits closely associated with glacial ice during deposition can contain relatively high concentrations of alkali-reactive aggregate. Such deposits include undifferentiated ice-contact deposits and eskers as well as outwash and delta features laid down close to the ice margin. In contrast, outwash features and associated glaciolacustrine deltas deposited by sediment-bearing meltwaters which have travelled long distances in meltwater valleys prior to deposition may contain relatively lower concentrations of alkali-reactive aggregate. The erosion of local bedrock, other than the Huronian Supergroup, in the meltwater valley would tend to dilute the concentration of alkali-reactive material carried by the meltwaters.

CONCLUDING REMARKS

As a result of this study, gravel pit operators in the Sudbury area have the option of obtaining aggregate that meets MTO concrete specifications from local, naturally occurring sand and gravel deposits, rather than hauling acceptable material over distances in excess of 80 km.

This paper is published with the approval of V.G. Milne, Director, Ontario Geological Survey.

REFERENCES

[1] Card, K.D. and Lumbers, S.B., Sudbury-Cobalt Sheet, Algoma, Manitoulin, Nipissing, Parry Sound, Sudbury, and Timiskaming Districts, Ontario Geological Survey, Map 2361, 1977.

[2] Giblin, P.E. and Leahy, E.J., Sault Ste. Marie-Elliot Lake Sheet, Algoma, Manitoulin and Sudbury Districts, Ontario Geological Survey, Map 2419, 1979.

[3] Dolar-Mantuani, L., Alkali-Silica-Reactive Rocks in the Canadian Shield, Highway Research Record No. 268, p.99-117, 1969.

[4] Grattan-Bellew, P.E., Study of Expansivity of a Suite of Quartzwackes, Argillites and Quartz Arenites, in Proceedings of 4th International Conference on the Effects of Alkalies in Cement and Concrete, Purdue University, Publication No. CE-MAT-1-78, p.113-140, 1978.

[5] Grattan-Bellew, P.E. and Rogers, C.A., National Research Council Studies of Alkali-Aggregate Reaction Systems and Test Methods, Reprint from International Workshop on Alkali-Aggregate Reactions in Concrete and Construction and Rehabilitation of Concrete Parking Structures, Montreal, Quebec, 20p., 1985.

[6] Rogers, C.A., Alkali-Aggregate Reactions, Concrete Aggregate Testing and Problem Aggregate in Ontario, A Review, Ontario Ministry of Transportation, Engineering Materials Office, Report EM-31, 5th Ed., 44p., 1985.

[7] Magni, E.R., Rogers, C.A. and Grattan-Bellew, P.E., The Influence of Alkali-Aggregate Reaction on Structures in the Vicinity of Sudbury, Ontario, in Proceedings, 7th International Conference on Alkali-Aggregate Reaction, edited by P.E. Grattan-Bellew, Noyes Publications, New Jersey, U.S.A., p.17-22, 1987.

[8] Magni, E.R. and Rogers, C.A., The Influence of the Alkali-Silicate Reaction on Structures in the Vicinity of Sudbury, Ontario, Ontario Ministry of Transportation, Engineering Materials Office, Report EM-81, 1987.

[9] Ontario Geological Survey, Aggregate Resources Inventory West of Sudbury, Ontario Geological Survey, Aggregate Resources Inventory Paper 140, 152p., 1987.

[10] Boissonneau, A.N., Surficial Geology, Algoma, Sudbury, Timiskaming and Nipissing, Ontario Department of Lands and Forests, Map S465, 1965.

[11] Boissonneau, A.N., Glacial History of Northeastern Ontario, 2: The Timiskaming-Algoma Area, in Canadian Journal of Earth Sciences, Volume 5, p.97-109, 1968.

[12] Burwasser, G.J., Quaternary Geology of the Sudbury Basin Area, Ontario Geological Survey, Report 181, 103p., 1979.

[13] Prest, V.K., "Quaternary Geology" in Geology and Economic Minerals of Canada, Geological Survey of Canada, Economic Geological Report, No. 1, 5th Ed., 838p., 1970.

[14] Harrison, J.E., Quaternary Geology of the North Bay-Mattawa Region, Geological Survey of Canada, Paper 71-26, 37p., 1972.

[15] Saarnisto, M., The Deglaciation History of the Lake Superior Region and its Climatic Implications, in Quaternary Research, Volume 4, p.316-339, 1974.

Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity; *Proceedings of a workshop held on March 19, 1990 at the American Concrete Institute Convention, Toronto; Published by: Ministry of Transportation, Ontario, Engineering Materials Report 92, March 1990.*

INTERLABORATORY STUDY OF THE NBRI RAPID TEST METHOD AND CSA STANDARDIZATION STATUS

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ABSTRACT

A rapid test method for detecting alkali-silica reactive aggregates was proposed by Oberholster and Davies of NBRI [1]. Testing in several Canadian laboratories on numerous aggregates appeared to support the applicability of this test method. Therefore, the CSA A5 subcommittee on alkali-aggregate reactions decided to draft a method based on the NBRI procedure and to undertake an interlaboratory test program to evaluate its precision and to evaluate the influence of cement alkali content an expansions obtained. Results of the interlaboratory test were positive and a second interlaboratory test is proposed.

INTRODUCTION

Because of the lengthy lead time required to adequately evaluate aggregate sources for potential alkali-aggregate reactivity, there is an urgent need for a reliable rapid test method. While numerous methods have been proposed, most have been found to be deficient or be limited in applicability to certain aggregate types. In 1986, Oberholster and Davies [1] published a new rapid test method for detecting alkali-silica reactive aggregates. It essentially involves crushing the aggregates and fabricating standard ASTM C 227 mortar bars. However, after the molds are stripped at one day, the bars are placed in water which is then heated up to 80°C. On the second day, the initial length readings are made and the bars are transferred to a 1N NaOH solution at 80°C where they are stored. Expansions are monitored for a period of only 14 days in the sodium hydroxide solution at which point the test is terminated. The expansions obtained in this rapid test are comparable or higher than those obtained after one year in the ASTM C 227 test (38°C at 100% humidity) [2].

Almost immediately after publication of Oberholster and Davies' paper, this test was being evaluated in Canada by a number of researchers on the CSA A5 Alkali-Aggregate Subcommittee, including Prof. M.A. Berube at Laval University, Dr. P.E. Grattan-Bellew at the Institute for Research in Construction in Ottawa, Mr. C.A. Rogers at the Ontario Ministry of Transportation (MTO) and the author. The results were very encouraging, and a CSA draft test method was written by the author together with C.A. Rogers. This draft method is attached as Appendix A. The author also simultaneously developed a similar Proposal Standard for ASTM which was accepted in 1989 and will be published [3].

In January, 1989, the CSA Subcommittee decided to initiate an interlaboratory test program with 3 cements of various alkali contents and 3 aggregates exhibiting different performances (innocuous, marginal and reactive). The purpose was to develop interlaboratory precision data on the expansions and to help define critical expansion limits.

EXPERIMENTAL

Materials

Cements (7 kg samples) were shipped by C. Bedard of Lafarge Canada Ltd.'s laboratory in Montreal. All laboratories received a high-alkali cement (Cement A) and a low-alkali cement (Cement B). For the third cement, each laboratory was shipped a different cement (Cement C → K) one of which was a silica fume blended cement (Cement K, approximately 8% silica fume). The compositions of these cements are given in Table 1.

The aggregates (15–17 kg samples) were shipped by C.A. Rogers from the MTO's stockpiles of aggregates at their laboratories in Toronto. The first was a reactive quarried limestone from near Ottawa (Spratt) which contained less than 5% chalcedony as the reactive component. The second was a carbonate sand from near Paris, Ontario (Paris) containing 0.2% leached chert. With the exception of minor popouts due to freezing of the chert, this aggregate has an excellent field performance record in concrete. The third aggregate was an innocuous crushed dolostone from the Niagara escarpment west of Toronto (Nelson).

Procedures Each laboratory followed the draft test method given in Appendix A and cast at least three mortar bars for each of the nine cement-aggregate combinations. All test results were reported to the author on a special reporting form for analysis of the data. The participating laboratories are listed in Table 2. From shipping to receipt of all results, the interlaboratory program only took 6 weeks.

RESULTS

The average 14 day (in NaOH solution) expansions obtained by each laboratory (coded) are given in Tables 3, 4 and 5 for the Spratt, Paris and Nelson aggregates respectively. Where they were recorded, mortar flows are also provided in these tables.

When the results of all nine laboratories are included, coefficients of variation ranged from 20 to 26% for the Spratt (Table 3), 21 to 47% for the Paris (Table 4), and 116 to 186% for the Nelson aggregate (Table 5). However, three of the laboratories gave consistently lower expansions: Both laboratories 6 and 9 were inexperienced with this test procedure and Laboratory 8 inadvertently used the wrong water/cement ratios. Therefore, statistics were also calculated in Tables 3, 4 and 5 with data from those three laboratories removed. Also, for the variable cement series, laboratory 5 was given the silica fume blended cement (Cement K),. An unusual feature of this test is that while the alkali content of the portland cement does not have a large influence on expansions [1], supplementary cementing materials or blended cements appear to suppress deleterious expansions [4]. (The reasons for this behaviour are still the subject of conjecture, therefore, at present this test is not recommended for evaluating the ability of blended cements to ameliorate reactive aggregates.) Therefore, the Cement K results were also removed in the second set of statistics in each of Tables 3, 4 and 5.

With these "outliers" removed, the coefficients of variation were dramatically improved: 7 to 12% (Spratt), 9 to 11% (Paris) and 46 to 63% (Nelson). For the innocuous Nelson aggregate, the average expansions were so low that the coefficients of variation are meaningless. It is interesting to note that for the reactive Spratt aggregate (Table 3), the use of different cements by each laboratory did not raise the coefficients of variation.

The average expansions using the low-alkali Cement B were not greatly lower than those using the high-alkali Cement A. For the Spratt aggregate the average difference in expansion between the two sets was only 8% while it was 27% for the less expansive Paris sand. While, the alkali content of the cement may have some effect on expansions it is certainly not the major effect that is observed in ASTM C 227 tests or in concrete.

Additional information obtained from each laboratory on the time taken to obtain readings from when the bar was removed from the 80°C solution, and on the range of the oven temperatures used is recorded in Table 6. It can be seen that none of the laboratories had a problem with obtaining a reading within the 15 second limit proposed in Section 9.2 of the draft method. however, some laboratories did not stay within the 15 $\pm$ 5 second range. Also, several laboratories reported that some mixes were either too wet or dry using the fixed w/c. However, with the fixed w/c, proportioning is much easier and less time consuming.

CONCLUSIONS AND RECOMMENDATIONS

1. The NBRI test method shows promise for adoption as a standard method by CSA.

2. The reproducibility of results appears to be very good when laboratories experienced with the procedures are involved.

3. The alkali content of the portland cement has only a minor effect on expansions. Therefore, this test can only be used to evaluate aggregates, rather than cement-aggregate combinations.

4. Because of concerns with using fixed water/cement ratios mortars (rather than the fixed flow used in ASTM C 227), a second interlaboratory program is proposed to determine which method gives the least variation.

REFERENCES

[1] Oberholster, R.E., and Davies, G., An Accelerated Method for Testing the Potential Alkali-Reactivity of Siliceous Aggregates. Cement and Concrete Research, 16, 181, 1986.

[2] Hooton, R.D. and Rogers, C.A., Evaluation of Rapid Test Methods for Detecting Alkali- Reactive Aggregates, Proceedings, 8th International Conference on Alkali-Aggregate Reaction, Kyoto, 439, 1989.

[3] ASTM C-9 Proposal P214, Proposed Test Method for Accelerated Detection of Potentially Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction, 4pp, 1990 Book of Standards, Vol. 04.02.

[4] Oberholster, R.E. and Davies, G., Use of the NBRI Accelerated Tests Evaluate the Effectiveness of Mineral Admixtures in Preventing the Alkali-Silica Reaction, Cement and Concrete Research, 17, 97, 1987.

Table 1
Compositions of Interlaboratory Cements*

CEMENT	Oxides (%)											Bogue Compounds*** (%)				BLAINE
	SiO_2	Al_2O_3	TiO_2	P_2O_5	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	LOI	C_3S	C_2S	C_3A	C_4AF	m^2/kg
A	20.00	5.56	0.21	0.09	1.89	62.99	1.36	0.10	1.24	3.83	2.71	53.5	17.0	11.5	5.8	385
B	22.79	4.24	0.14	0.13	2.58	63.84	1.44	0.16	0.36	2.39	2.36	47.3	29.3	6.9	7.8	364
C	20.04	5.64	0.21	0.09	1.89	62.95	1.31	0.11	1.22	4.79	0.98	49.7	20.0	11.8	5.8	592
D	21.21	4.18	0.20	0.19	2.90	61.55	2.59	0.35	0.96	3.43	2.58	45.1	26.8	7.2	8.8	389
E	20.90	4.87	0.26	0.09	2.60	64.50	2.08	0.16	0.93	2.39	1.46	60.2	14.6	8.6	7.9	354
F	22.34	2.95	0.18	0.07	4.53	65.90	0.94	0.12	0.46	2.38	0.85	65.4	14.7	0.2	13.8	390
G	21.40	4.44	0.19	0.07	3.30	65.20	0.83	0.19	0.63	2.77	1.01	60.0	16.2	6.2	10.0	356
H	22.10	5.62	0.30	0.10	2.20	63.22	1.13	0.06	0.42	3.42	1.62	39.9	33.2	11.2	6.7	384
I	19.80	5.38	0.40	0.10	2.63	63.42	2.86	0.14	0.48	2.93	1.99	55.9	14.9	11.1	8.2	358
K**	26.77	3.95	0.15	0.21	2.94	58.46	2.04	0.24	0.76	3.64	1.70	–	–	–	–	518

* Cements analysed by Lafarge Canada Ltd.using X-ray fluorescence.
** Blended cement containing 8% silica fume.
*** Bogue calculated as per ASTM C150 ie: $R_2O_3 = Al_2O_3 + TiO_2 + P_2O_5$.

Table 2
List of Nine Participating Laboratories

- Institute for Research in Construction, Ottawa
- Canadian National Railways , Montreal
- Ecole Polytechnique, Montreal
- Ontario Ministry of Transportation, Toronto
- Quebec Ministry of Transport, Quebec
- Laval University, Quebec
- Laboratoire de Beton Ltée, Montreal
- Ontario Hydro, Toronto
- University of Toronto, Toronto

Table 3
NBRI 14 Day Test Expansions for Reactive Spratt Aggregate (w/c = 0.50)

LABORATORY CODE	CEMENT A		CEMENT B		VARIABLE		CEMENT CODE
	EXP'N (%)	FLOW	EXP'N (%)	FLOW	EXP'N (%)	FLOW	
1	.508	98	.400	100	.508	100	E
2	.3490	84	.3533	95	.4596	88	I
3	.4127	110	.4323	113	.4293	90	C
4	.4160	107	.4155	99	.4584	97	G
5	.4173	116***	.3973	120***	.2103	113	K****
6*	.2743	–	.2403	–	.3000	–	H
7	.4288	too fluid	.3391	too fluid	.4319	too fluid	H
8**	.2783	–	.2617	–	.2937	–	F
9*	.2920	–	.2927	–	.3230	–	D
ALL LABS							
MEAN ($\bar{X}_n$)	(n=9)	0.3767	(n=9)	0.3480	(n=9)	0.3794	
STD. DEVIAITON ($\sigma - 1$)		0.079		0.0698		0.0999	
COEFF. OF VARIAITON		21.00%		20.07%		26.34%	
OUTLIERS REMOVED		6, 8, 9		6, 8, 9		5, 6, 8, 9	
MEAN ($\bar{X}_n$)	(n=6)	0.4220	(n=6)	0.3896	(n=6)	0.4574	
STD. DEVIATION ($\sigma - 1$)		0.0508		0.03614		0.0316	
COEFF. OF VARIATION		12.04%		9.28%		6.92%	

* labs 6 and 9 were relatively inexperienced with this test method.
** lab 8 used w/c = 0.44 inadvertantly.
*** 10 drops only.
**** silica fume blended cement.

Table 4
NBRI 14 Day Test Expansions for Marginal Paris Sand (w/c = 0.44)

LABORATORY CODE	CEMENT A		CEMENT B		VARIABLE		CEMENT CODE
	EXP'N (%)	FLOW	EXP'N (%)	FLOW	EXP'N (%)	FLOW	
1	.178	105	.163	117	.182	116	E
2	.1570	88	.1280	94	.2063	91	I
3	.1877	106	.1450	116	.1560	97	C
4	.1876	117	.1472	109	.1957	93	G
5	.1603	107***	.1263	122***	.0476	112	K****
6*	.1130	–	.1010	–	.0573	–	H
7	.2129	–	.1412	–	.1985	–	H
8**	.1103	–	.0770	–	.0837	–	F
9*	–	–	–	–	–	–	
ALL LABS MEAN ($\bar{X}_n$)	(n=8)	0.1634	(n=8)	0.1286	(n=8)	0.1409	
STD. DEVIAITON ($\sigma - 1$)		0.0364		0.0277		0.0671	
COEFF. OF VARIAITON	22.25%		21.57%		47.64%		
OUTLIERS REMOVED	6, 8		6, 8		5, 6, 8		
MEAN ($\bar{X}_n$)	(n=6)	0.1806	(n=6)	0.1418	(n=5)	0.1877	
STD. DEVIATION ($\sigma - 1$)		0.206		0.1356		0.198	
COEFF. OF VARIATION	11.40%		9.57%		10.53%		

* labs 6 and 9 were relatively inexperienced with this test method (Lab 9 did not complete this set of tests).
** lab 8 used w/c = 0.50 inadvertantly.
*** 10 drops only.
**** silica fume blended cement.

Table 5

NBRI 14 Day Test Expansions for Innocuous Nelson Stone (w/c = 0.50)

LABORATORY CODE	CEMENT A		CEMENT B		VARIABLE		CEMENT CODE
	EXP'N (%)	FLOW	EXP'N (%)	FLOW	EXP'N (%)	FLOW	
1	.007	104	.016	110	.012	112	E
2	.0140	93	.0137	104	.0137	92	I
3	.0087	112	.0087	120	.0043	97	C
4	.0143	118	.0147	107	.0066	100	G
5	.0130	140	.0123	140***	.0117	140****	K****
6*	-.0133	–	.0133	–	-.0157	–	H
7	.0285	too fluid	.0299	too fluid	.0270	too fluid	H
8**	-.0013	–	.0026	–	.0047	–	F
9*	–	–	–	–	–	–	
ALL LABS MEAN ($\bar{X}_n$)	(n=8)	0.0089	(n=8)	0.0106	(n=8)	0.0069	
STD. DEVIAITON ($\sigma - 1$)		0.0123		0.0124		0.0128	
COEFF. OF VARIAITON	138.0%		116.6%		185.9%		
OUTLIERS REMOVED	6, 8		6, 8		6, 8		
MEAN ($\bar{X}_n$)	(n=6)	0.0143	(n=6)	0.0159	(n=6)	0.0126	
STD. DEVIATION ($\sigma - 1$)		0.0076		0.0073		0.0079	
COEFF. OF VARIATION	53.07%		45.97%		62.96%		

* labs 6 and 9 were relatively inexperienced with this test method (Lab 9 did not complete this set of tests).

** lab 8 used w/c = 0.44 inadvertantly.

*** 18 drops only.

**** silica fume blended cement.

Table 6
Additional Infomation

LABORATORY CODE	RANGE OF TIMES TO OBTAIN READINGS* (sec.)	RANGE OF OVEN TEMP (°C)
1	8–12	78–82
2	5–7	78–82
3	10–14	79–80
4	6	78–82
5	10	76–84
6	2–5	81
7	10–12	77–85**
8	5	79–81
9	15	78–82

* Time from when each bar was removed from the 80°C NaOH solution until measurement was recorded.

** Three different ovens, each with a smaller temp. range.

APPENDIX A

R.D. HOOTON AND C. ROGERS
DRAFT #3: January 25, 1990

CSA CAN3-A23.2-XXC

Standard Test Method for Accelerated Detection of Potentially Deleterious Expansion of Mortar Bars Due to Alkali-Silica Reaction

1. Scope:

1.1 This test method is for detecting within 16 days the potential for deleterious expansion of mortar bars due to the alkali-silica and slow/late-expanding alkali silicate/silica reactions.

1.2 Alkalies participating in the expansive reactions usually are derived from the cement; under some circumstances they may be derived from other constituents of the concrete or from external sources. Three types of alkali reactivity of aggregates are recognized: (1) an alkali-silica reaction involving certain siliceous rocks, minerals, and natural or artificial glasses (see CAN3-A23.1, Appendix B2.2), (2) a slow/late-expanding alkali-silicate/silica reaction (see CAN3-A23.1, Appendix B2.3), and (3) an alkali-carbonate reaction involving dolomite in certain calcitic dolostones and dolomitic limestones. The method is not recommended as a means to detect the latter reaction because expansions produced in the mortar-bar test by the alkali-carbonate reaction are generally much less than those produced by the alkali-silica reaction for combinations having equally harmful effects in service. The concrete prism expansion test (CAN3-A23.2-14A) is the only satisfactory method of determining expansivity of alkali-carbonate reactive aggregates.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific precautionary statement is given in 5.3.1.

2. Referenced Documents:

2.1	CSA Standards:	CAN3-A5-M88	Portland Cements
		CAN3-A23.2-14A	Alkali-aggregate Reaction
		CAN3-A23.1-M77	Appendix B (Supplement No. 2 - 1986)
2.2	CSGB Standards:	8-GP-2M	Sieves, Testing, Woven Wire, Metric
2.3	ASTM Standards:	C 227	Test Method for Potential Alkali Reactivity of Cement - Aggregate Combinations (Mortar Bar Method)
		C 490	Specification for Apparatus for Use in Measurement of Length Change of Hardened Cement Paste, Mortar, and Concrete.

3. Significance and Use:

3.1 This test method provides a means of detecting the potential of an aggregate used in concrete for undergoing alkali-silica or slow/late-expanding alkali silicate/silica reactions and consequent potentially deleterious expansion. It is based on the NBRI Accelerated Test Method[1,2,3]. It provides an alternative method to ASTM C 227 and may be especially useful for aggregates that react slowly or produce expansion late in the reaction.

Note 1 - Because the specimens are exposed to a NaOH solution, the alkali content of the cement is not a significant factor in affecting expansions. However, the method has been found to be suitable for determining the ability of a supplementary cementing material to control the expansion[2]. However, further research is required before this test is recommended for evaluating supplementary cementing materials.

3.2 When expansions greater than 0.10% are developed within 16 days from casting, it is strongly recommended that supplementary information be developed to confirm that the expansion is actually due to alkali reactivity (see Appendix B of CAN3-A23.1).

3.3 When it has been concluded from the results of tests performed using this method and supplementary information that a given aggregate should be considered potentially deleteriously reactive, additional studies possibly using alternate test methods, may be appropriate to develop information on the potential reactivity of other combinations containing the same cement with other aggregates, or the same cement-aggregate combination with supplementary cementing materials.

4. Apparatus:

4.1 The apparatus shall conform to ASTM Specification C 490, except as follows:

4.2 *Sieves* - Square hole, woven-wire cloth sieves, shall conform to CGSB Standard 8-GP-2M.

4.3 *Mixer, Paddle, and Mixing Bowl* - Mixer, paddle, and mixing bowl shall conform to the requirements of CAN3-A5-M88, Section 8.4.5, except that the clearance between the lower end of the paddle and the bottom of the bowl shall be 5.1 ± 0.3 mm.

4.4 *Tamper and Trowel* - The tamper and trowel shall conform to CAN3-A5-M88 Sections 8.4.8 and 8.4.9.

4.5 *Containers* - The containers must be of such a nature that the bars can be totally immersed in either the water or 1N NaOH solution. The containers must be made of material that can withstand prolonged exposure to 80°C and must be inert to a 1N NaOH solution. The containers must be so constructed that when used for storing specimens the loss of moisture is prevented by tight-fitting covers or by sealing or by - both. The bars in the solution must be placed and supported so that the solution has access to the whole of the bar; therefore, it should be ensured that the specimens do not touch the sides of the container or each other. The specimens, if stood upright in the solution, cannot be supported by the metal gauge stud.

Note 2 - The caustic NaOH solution will corrode glass or metal containers.

Note 3 - A covered container that has been found to be acceptable for this purpose is sold by Rubbermaid as a microwave-proof food storage container,

4.6 *Oven* - A convection oven with temperature control maintaining 80° ± 2°C.

5. **Reagents:**

5.1 *Sodium Hydroxide (NaOH)* - USP or technical grade may be used, provided it is established that it is of sufficiently high purity to permit its use without lessening the accuracy of the determination. When tests are made that are expected to produce results that are close to an acceptance-rejection value, it is recommended that reagent grade chemicals be used.

5.2 *Purity of Water* - Unless otherwise indicated, references to water shall be understood to mean distilled water.

5.3 *Sodium Hydroxide Solution* - Each litre of solution shall contain 40.0 g of NaOH dissolved in 900 ml of water, and shall be diluted with additional distilled or deionized water to obtain 1.0L of solution. The volume proportion of sodium hydroxide solution to mortar bars in a storage container shall be 4 ± 0.5 volumes of solution to 1 volume of mortar bars.

Note 4 - The volume of a mortar bar may be taken as 184 mL.

5.3.1 *Precaution* - Before using NaOH review (1) the safety precautions for using NaOH, (2) first aid for burns, and (3) the emergency response to spills, as described in the manufacturer's Material Safety Data Sheet or other reliable safety literature. NaOH can cause very severe burns and injury to unprotected skin and eyes. Suitable personal protective equipment should always be used. These should include full-face shields, rubber aprons, and gloves impervious to NaOH. Gloves should be checked periodically for pin holes.

6. **Temperature and Humidity:**

6.1 The temperature of the molding room and dry materials shall be maintained at not less than 20°C and not more than 27.5°C. The temperature of the mixing water and of the moist closet or moist room shall not vary from 23°C by more than 2°C.

6.2 The relative humidity of the molding room shall be not less than 50%. The moist closet or room shall conform to CAN3-A5-M83, Clause 8.3.2.

6.3 The storage oven in which the specimens are stored in the containers shall be maintained at a temperature that shall not vary from 80°C by more than 2°C.

7. **Selection and Preparation of Materials:**

7.1 *Selection of Aggregate* - Materials proposed for use as fine aggregate in concrete shall be processed as described in 7.2 with a minimum of crushing. Materials proposed for use as coarse aggregate in concrete shall be processed by crushing to produce as nearly as practical a graded product from which a sample can be obtained The sample shall have the grading as prescribed in 7.2 and be representative of the composition of the coarse aggregate as proposed for use.

7.1.1 When a given quarried material is proposed for use, both as coarse and as fine aggregate, it will be tested only by selection of an appropriate sample crushed to the fine aggregate sizes, unless there is reason to expect that the coarser size fractions have a different composition than the finer sizes and that these differences might significantly affect expansion due to reaction with the alkalies in cement. In this case the coarser size fractions should be tested in a manner similar to that employed in testing the fine aggregate sizes.

7.1.2 Coarse aggregate crushed to sand size may give increased expansion, owing to the increased surface exposed upon crushing. Therefore, if coarse aggregate tested by this method produces an excessive amount of expansion, the material shall not be classed as objectionably reactive with alkali unless tests of concrete specimens confirm the findings of the tests of the mortar.

7.2 *Preparation of Aggregate* - All aggregates to which this method is applied shall be graded in accordance with the requirements prescribed in Table 1. Aggregates in which sufficient quantities of the sizes specified in Table 1 do not exist shall be crushed until the required material has been produced. In the case of aggregates containing insufficient amounts of one or more of the larger sizes listed in Table 1, and if no larger material is available for crushing, the first size in which sufficient material is available shall contain the cumulative percentage of material down to that size as determined from the grading specified in Table 1. When such procedures are required, special note shall be made thereof in the test report. After the aggregate has been separated into the various sieve sizes, each size shall be washed with a water spray over the sieve to remove adhering dust and fine particles from the aggregate. (Note 5). The portions retained on the various sieves shall then be dried and, unless used immediately, each such portion shall be stored individually in a clean container provided with a tight-fitting cover.

TABLE 1: GRADING REQUIREMENTS

Sieve Size				Mass %
Passing		**Retained on**		
5	mm	2.5	mm	10
2.5	mm	1.25	mm	25
1.25	mm	630	µm	25
630	µm	315	µm	25
315	µm	160	µm	15

Note 5 - Tests have shown that mortar bar expansions obtained in this method are less susceptible than in ASTM Method C 227 to variability if the aggregates are not washed.

7.3 ***Selection and Preparation of Cement:***

7.3.1 *Reference Cement* - The portland cement used shall meet the requirements of CAN3-A5-M83.

Note 6 - Since the bars are immersed in NaOH solution, the alkali content of the cement has a negligible influence on expansion.

7.3.2 *Preparation of Cement* - Cement for use in this test shall be passed through a 1.25 mm sieve to remove lumps before use.

8. **Preparation of Test Specimens:**

8.1 *Number of Specimens* - Make at least three test specimens for each cement-aggregate combination.

8.2 *Preparation of Molds* - Prepare the specimen molds in accordance with the requirements of ASTM Specification C 490 except, the interior surfaces of the mold shall be covered with a release agent. A release agent will be acceptable if it serves as a parting agent without affecting the setting of the cement and without leaving any residue that will inhibit the penetration of water into the specimen.

Note 7 - TFE-fluorocarbon tape complies with the requirements for a mold release agent.

8.3 *Proportioning of Mortar* - Proportion the dry materials for the test mortar using 1 part of cement to 2.25 parts of graded aggregate by mass. The quantities of dry materials to be mixed at one time in the batch of mortar for making three specimens shall be 440g of cement and 990g of aggregate made up by re-combining the portions retained on the various sieves (see 7.2) in the grading prescribed in Table 1. For natural fine aggregates, use a water to cement ratio equal to 0.44 by mass. For coarse aggregates or manufactured sands, use a water to cement ratio equal to 0.50 by mass.

Note 8 - Ruggedness tests indicated that mortar bar expansions were less variable at a fixed water to cement ratio than when gauged to a constant flow.[3] The water to cement ratios selected should give acceptable workability in most cases.[4]

8.4 *Mixing of Mortar* - Mix the mortar in accordance with the requirements of Method CAN3-A5-M88, Section 7.5.

8.5 *Molding of Test Specimens* - Start molding specimens with a total elapsed time of not more than 2 min and 15 s after completion of the original mixing of the mortar batch. Fill the molds with two approximately equal layers, each layer being compacted with the tamper. Work the mortar into the corners, around the gauge studs, and along the surfaces of the mold with the tamper until a homogeneous specimen is obtained. After the top layer has been compacted, cut off the mortar flush with the top of the mold and smooth the surface with two strokes of the trowel.

9. Storage and Measurement of Test Specimens

9.1 *Initial Storage and Measurement* - After each mold has been filled, place the mold immediately in the moist cabinet or room. The specimens shall remain in the molds for 24 ± 2 h. Then remove them from the molds and, while being protected from loss of moisture, properly identify. The initial and all subsequent measurements shall be made and recorded to the nearest 0.002 mm. Place the specimens of each aggregate sample in a storage container with sufficient tap water to totally immerse them. Seal and place the containers in an oven or room at 80°C ± 2°C for a period of 24 hours.

9.2 *Zero Measurements* - For the zero measurement of the specimens, the containers shall be removed from the oven one at a time. Other containers are only removed once the bars in the first container have been measured and returned to the oven. Remove the bars one at a time from the water and dry their surface with a towel paying particular attention to the two metal gauge measuring studs. The zero measurement of each prism is done immediately after drying and the reading is taken as soon as the bar is in position. The process of drying and measuring is to be completed within 15 ± 5 seconds of removing the specimen from the water. After measurement, the specimen is left on a towel until the remainder of the bars have been measured, after which they are all placed in a separate container with sufficient 1N NaOH, at 80° ± 2°C for the samples to be totally immersed. The container is then sealed and returned to the oven and the process repeated for the remaining containers.

Note 9 - The comparator bar should be measured prior to each set of specimens since the heat from the mortar bars may cause the length of the comparator to change.

9.3 *Subsequent storage and measurement.* Subsequent measurement of the specimens is undertaken periodically, with at least three intermediate readings, for 14 days after the zero reading, at approximately the same time each day. The measuring procedure is identical to that described in 9.2 except that the specimens are returned to their own container after measurement.

10. Calculation:

10.1 Calculate the difference between the zero length of the specimen and the length at each period of measurement to the nearest 0.001 per cent of the effective gauge length and record as the expansion of the specimen for that period. Report the average expansion of the four specimens of a given cement-aggregate combination to the nearest 0.01 per cent as the expansion for the combination for a given period.

11. Report:

11.1 The report shall include the following:

11.1.1 Type and source of aggregate,

11.1.2 Type and source of portland cement,

11.1.3 Alkali content of cement as percent potassium oxide (K_2O), sodium oxide (Na_2O), and calculated sodium oxide (Na_2O) equivalent,

11.1.4 Average length change in percent at each reading of the specimens,

11.1.5 Any relevant information concerning the preparation of aggregates, including the grading of the aggregate when it differs from that given in 7.2,

11.1.6 Any significant features revealed by examination of the specimens during and after test,

11.1.7 Amount of mixing water expressed as mass percent to cement, and

11.1.8 Type, source, proportions and chemical analyses, including Na_2O and K_2O, of any supplementary cementing materials employed in the tests.

11.1.9 A graph of the length change data from the time of the zero reading to the end of the 16 day period.

12. Interpretation of Results:

12.1 When the mean expansion of the test specimens exceeds 0.20% at 16 days from casting (14 days from zero reading), it is indicative of potentially deleterious expansion. This value has been determined from tests of aggregates with known field performance. It is strongly recommended that supplementary information be developed to confirm that the expansion is actually due to alkali reactivity (see Appendix B of CAN3-A23.1).

12.2 When the mean expansion of the test specimens is less than 0.10% at 16 days after casting, it is indicative of innocuous behaviour[5].

12.3 Where the mean expansion of the test specimens is above 0.10% and less than 0.20% at 16 days from casting, the results are as yet inconclusive. It is strongly recommended that supplementary information be developed as discussed in CAN3-A23.1, Appendix B.

13. Precision and Bias:

13.1 Precision data will have to be developed.

13.2 *Bias* - Since there is no accepted reference material for determining the bias of this test method, no statement on bias is being made.

References:

1. R.E. Oberholster and G. Davies, "An Accelerated Method for Testing the Potential Alkali Reactivity of Siliceous Aggregates, Cement and Concrete Research, Vol. 16, pp. 181-189, 1986.

2. G. Davies and R.E. Oberholster, "Use of the NBRI Accelerated Test to Evaluate the Effectiveness of Mineral Admixtures in Preventing the Alkali-Silica Reaction", Cement and Concrete Research, Vol. 17, pp. 97-107, 1987.

3. G. Davies and R.E. Oberholster, "An Interlaboratory Test Programme on the NBRI Accelerated Test to Determine the Alkali-Reactivity of Aggregates", National Building Research Institute, CSIRO, Special Report BOU 92-1987, Pretoria, RSA, 16 pp., 1987.

4. C. Rogers, Ontario Ministry of Transportation and Communications, Personal Communication, Tabulation of mean w/c for acceptable flow of mortar bars with fine and crushed-coarse aggregates, Oct. 25, 1988.

5. R.D. Hooton and C.A. Rogers, "Evaluation of Rapid Test methods for Detecting Alkali-reactive Aggregates", Proceedings, 8th International Conference on Alkali-Aggregate Reaction, pp. 439-434, 1989.